

# SRI International

Progress Report • March 1, 1993

## REPORT ON CHEMICAL ANALYSES OF PROVIDED SAMPLES

Christopher H. Becker  
Molecular Physics Laboratory

SRI Project 3557  
Contract No. 1-1EH-46755  
MP 93-044

Prepared for:

NASA Marshall Space Flight Center  
Huntsville, AL 35812

Attn: Roger Linton  
Mail Stop EH15

Approved:

Donald J. Eckstrom, Director  
Molecular Physics Laboratory

David M. Golden  
Vice President  
Physical Sciences Division

*1N-25 OR*  
*16*  
N93-27077  
Unclas  
0163577

G3/25

(NASA-CR-193211) CHEMICAL ANALYSES  
OF PROVIDED SAMPLES Final Report  
(SRI International Corp.) 40 p

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
<small>Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20603</small>				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE March 1993	3. REPORT TYPE AND DATES COVERED Progress Report		
4. TITLE AND SUBTITLE REPORT ON CHEMICAL ANALYSES OF PROVIDED SAMPLES			5. FUNDING NUMBERS	
6. AUTHOR(S) Christopher H. Becker				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) SRI International 333 Ravenswood Avenue Menlo Park, CA 94025			8. PERFORMING ORGANIZATION REPORT NUMBER PYU 3557 MP 93-044	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) NASA Marshall Space Flight Center Huntsville, AL 35812			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  Two batches of samples were received and chemical analysis was performed of the surface and near surface regions of the samples by the surface analysis by laser ionization (SALI) method. The samples included four one-inch optics and several paint samples. The analyses emphasized surface contamination or modification. In these studies, pulsed sputtering by 7 keV Ar <sup>+</sup> and primarily single-photon ionization (SPI) by coherent 118 nm radiation (at ~5 x 10 <sup>5</sup> W/cm <sup>2</sup> ) were used. For two of the samples, also multiphoton ionization (MPI) at 266 nm (~5 x 10 <sup>11</sup> W/cm <sup>2</sup> ) was used.  Most notable among the results was the silicone contamination on Mg <sub>2</sub> mirror #28-92, and that the LDEF paint sample had been enriched in K and Na and depleted in Zn, Si, B and organic compounds relative to the control paint.				
14. SUBJECT TERMS EDEF, surface analysis, surface contamination, space environmental effects			15. NUMBER OF PAGES 39	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	

The two batches of samples were received and chemical analysis was performed of the surface and near surface regions of the samples by the surface analysis by laser ionization (SALI) method. The analyses thus emphasize surface contamination or modification. SALI uses nonselective photoionization of sputtered or desorbed atoms and molecules above but close ( $\sim 1$  mm) to the surface, followed by time-of-flight (TOF) mass spectrometry. In these studies, pulsed sputtering by 7 keV  $\text{Ar}^+$  and primarily single-photon ionization (SPI) by coherent 118 nm radiation (at  $\sim 5 \times 10^5 \text{ W/cm}^2$ ) were used. For two of the samples, also multiphoton ionization (MPI) at 266 nm ( $\sim 5 \times 10^{11} \text{ W/cm}^2$ ) was used. SPI is designed primarily for its ability to obtain molecular information, whereas MPI is used primarily for elemental and small molecule information. For MPI, we generally prefer a higher laser intensity to achieve saturation of even high ionization potential elements, but geometric constraints prevented tighter focusing for these analyses; absolute concentrations would require calibration, but changes in relative signals do quantitatively reflect changes in relative concentrations.

While SPI by 118 nm (10.5 eV) light is considered a generally "soft" (nonfragmenting) form of radiation, sputtering causes fragmentation and also produces internally hot molecules which photofragment relatively easily compared to thermal sources of molecules. Therefore, the low mass regions of the mass spectra contain a good deal of molecular fragment information.

The samples included four 1 inch diameter optics, labeled UV-PG1 (a transparent substrate), LDEF mirror CM01-52,  $\text{MgF}_2$  mirror 28-92, and the control  $\text{MgF}_2$  mirror S1. Additionally, there were two YB-1 paint samples, the LDEF sample #02-35 and a control. These two paint samples were on metal backings which were too large to pass through the sample introduction region. Therefore small chips of the paints were used; it was only practical to obtain the chips from the edges of the samples, otherwise extensive damage to the samples would have resulted. It was for these two YB-1 paint samples that both SPI and MPI were used.

These analyses began with so-called static conditions (much less than one monolayer erosion). For samples that have been exposed to normal air, a monolayer or so of adventitious organic contamination is invariably found, as for these samples. (The ion-pumped ultrahigh vacuum analysis chamber and turbomolecular-pumped sample introduction region were baked just prior to these studies to minimize any contamination from the analysis system itself.) Therefore in studying the resultant mass spectra, it is necessary to distinguish between this general type of contamination and mass signals which are particular to the given sample. To a large degree this can be done by comparing the mass spectra for the various samples under static conditions and identifying the common components. Without going into excessive detail, for the 118 nm photoionization, there are numerous organic fragments (some of the more notable peaks being  $m/z$

= 15, 42, 52, 66, 78, and 91) which are found on essentially all samples. After looking over the spectra in some detail, the signals from the uninteresting adventitious organics will become apparent and can be distinguished from signals particular to each sample.

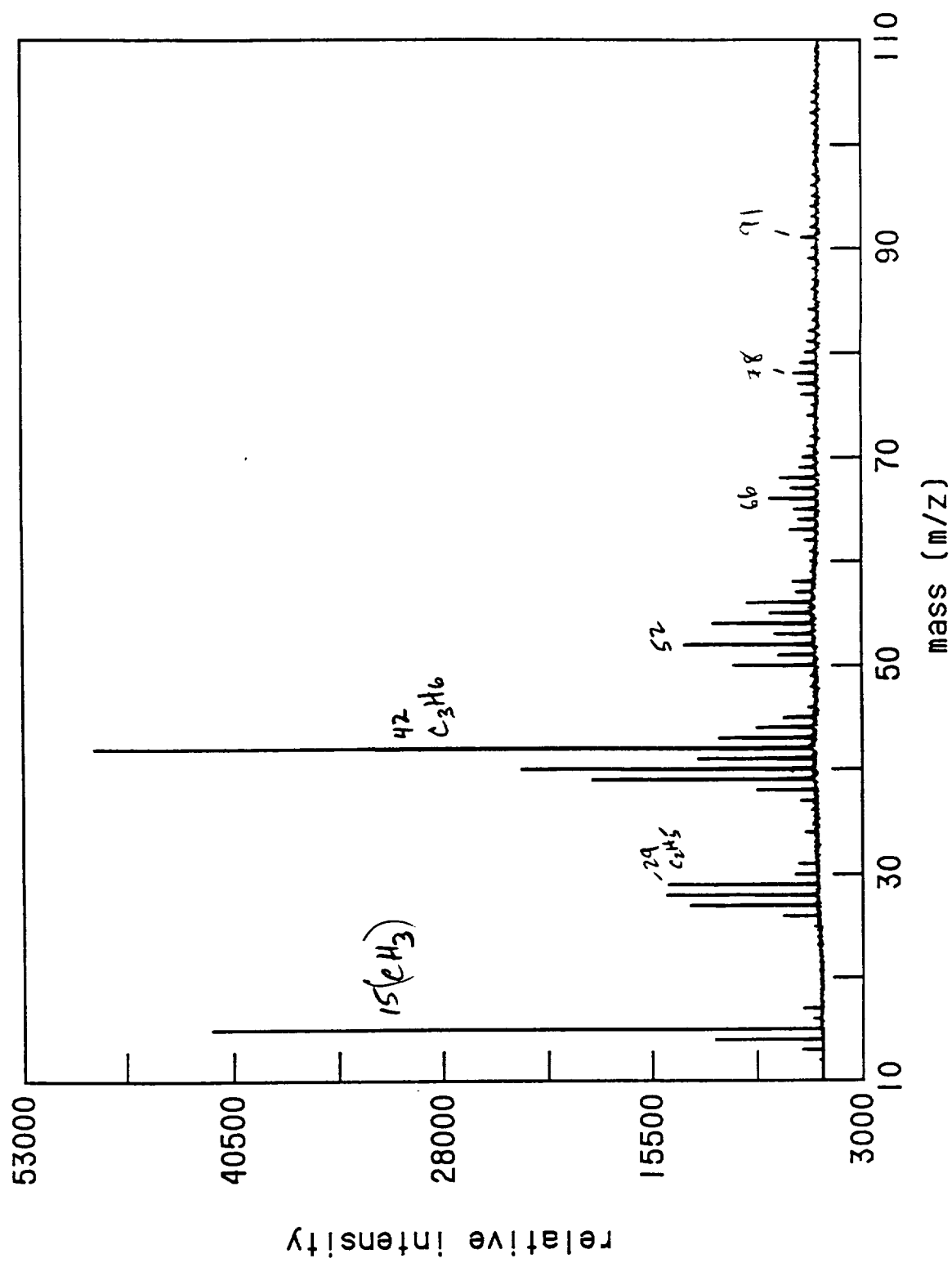
Some other comments on the mass spectra are appropriate. The signals from the microchannel plate detector were recorded in analog fashion by a 100 MHz transient digitizer; thus the voltage signals are given as "relative intensity" and not as ion counts. Some of the samples were somewhat problematic due to charging under the charged  $\text{Ar}^+$  beam; occasionally an electron gun was used to help neutralize the sample. Some of the baselines have a wavy background component which is due to secondary ions from the pulsed ion beam; these normally are rejected electrostatically, but the signals can originate from metastable molecular ions and from when the surface accumulates a net negative charge.

Some general comments on the analyses now follow. The mass spectra often contain a wealth of information that can be appreciated by some detailed study of the mass spectra, but we wish only to point out some of the more obvious facts in this text. Some notes on the experimental conditions and mass peak labelings (and some of their chemical assignments) are found on the spectra themselves. We can discuss mass assignments further if desired. The amount of erosion labeled is only very approximate; more specific is the amount of ion dose; for the notes on the figures, 1 monolayer (ML) corresponds to an  $\text{Ar}^+$  dose of  $5 \times 10^{14}/\text{cm}^2$  although surface charging can sometimes make the estimate of the ion dose somewhat uncertain. Thus amounts of erosion should be considered only qualitative. File names are printed to identify each spectra, e.g. FE2317.

For the four 1 inch diameter optics, only the  $\text{MgF}_2$  mirror #28-92 showed significant contamination. For this sample, peaks at  $m/z$  73, 147, 207, 221, 281, and others are clear indications of silicone (siloxane); chemical compositions corresponding to these masses can be provided if you are interested.

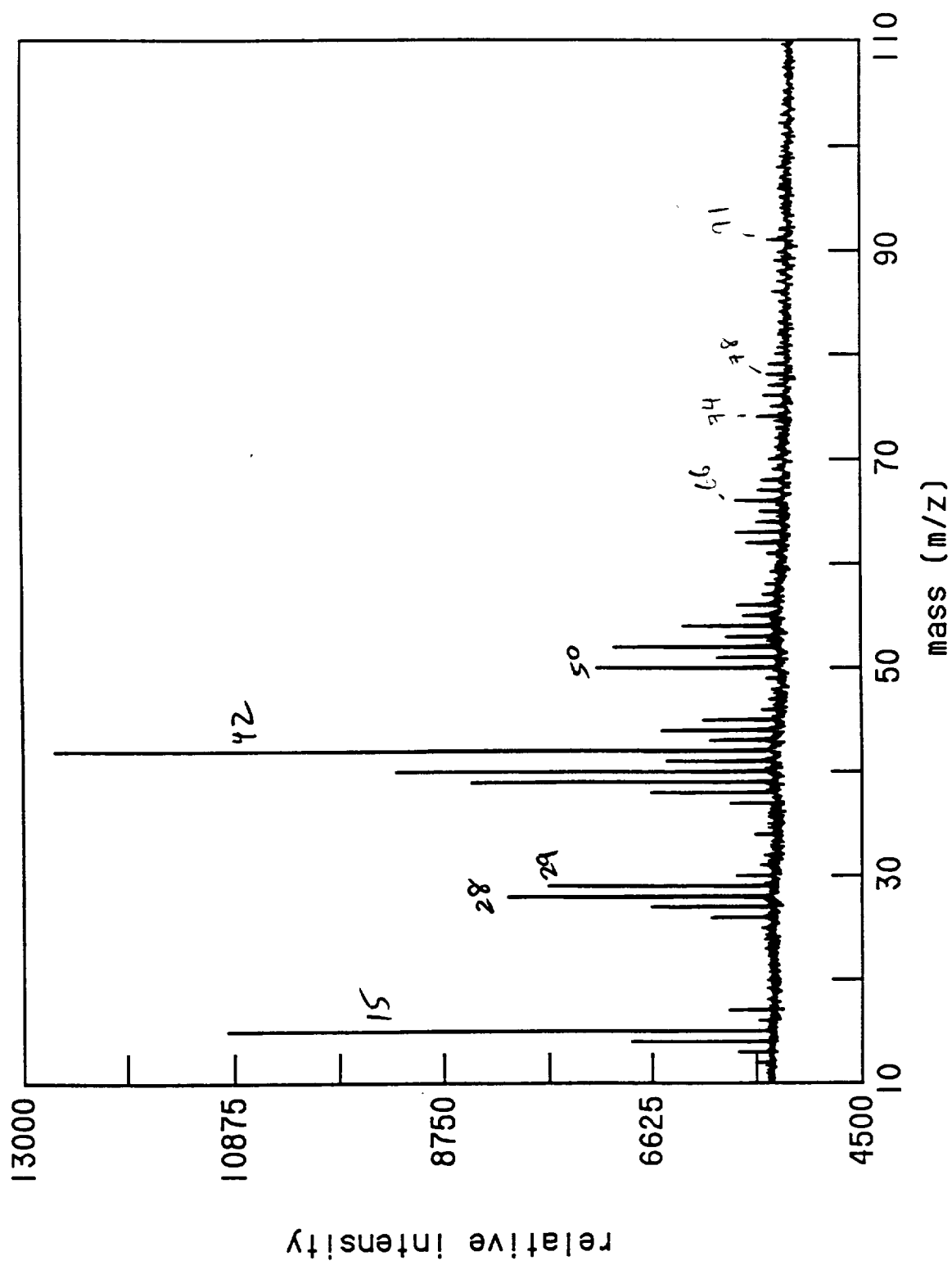
With regard to the two YB-1 paint samples, the 118 nm photoionization mass spectra show a loss in the polymeric/organic species for the LDEF sample relative to the control sample (e.g. see the series of peaks between 100 and 160 amu). The spectra taken with 266 nm (so labeled) show some differences in the inorganic chemistry of the surface region. For example, the control sample shows more Zn, Si, and B and less of the alkalis K and Na relative to the LDEF 02-35 sample.

We would be happy to discuss any of these results further.



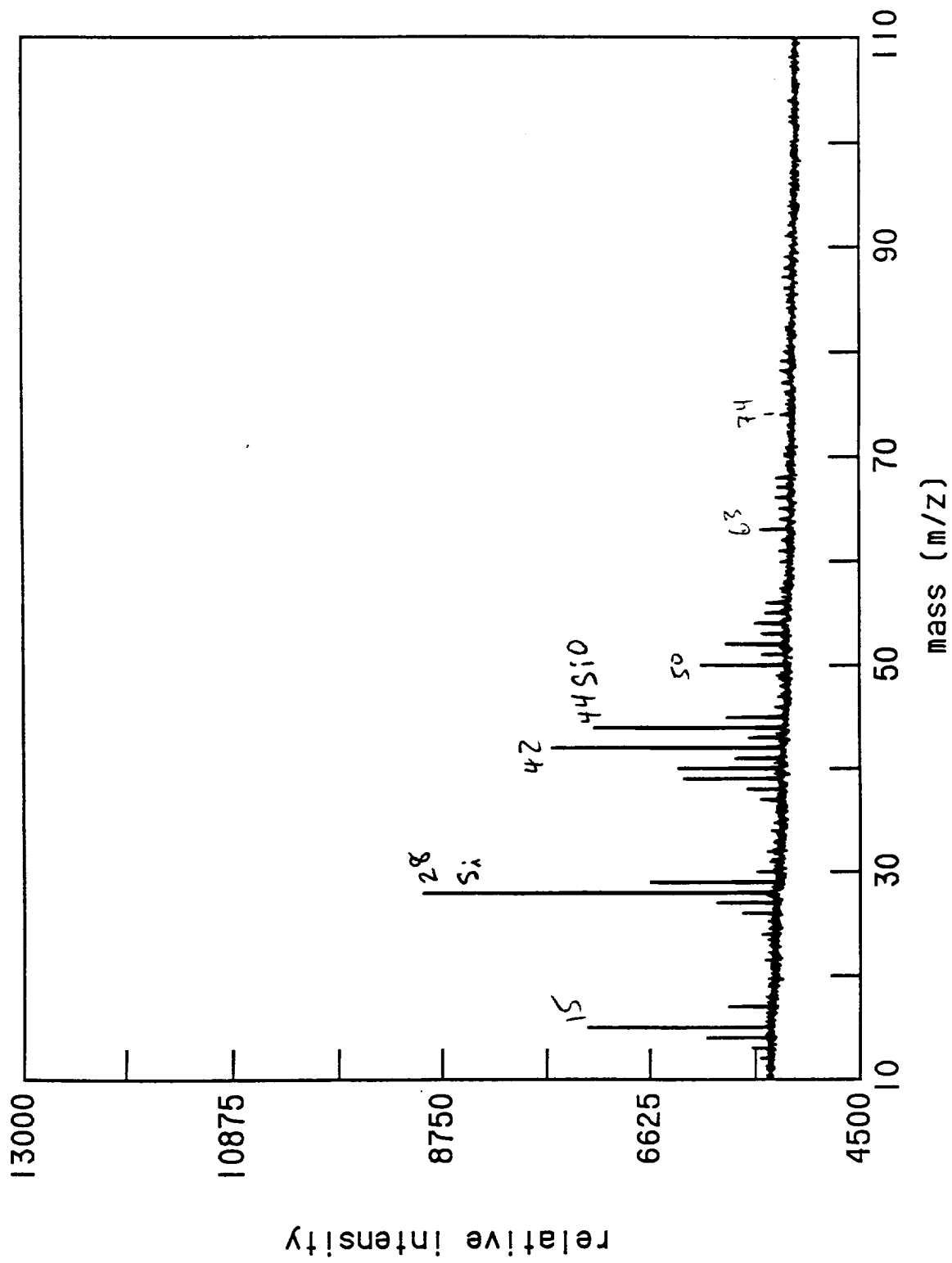
LDEF mirror CM01-52 fe2324 surface

*static conditions*



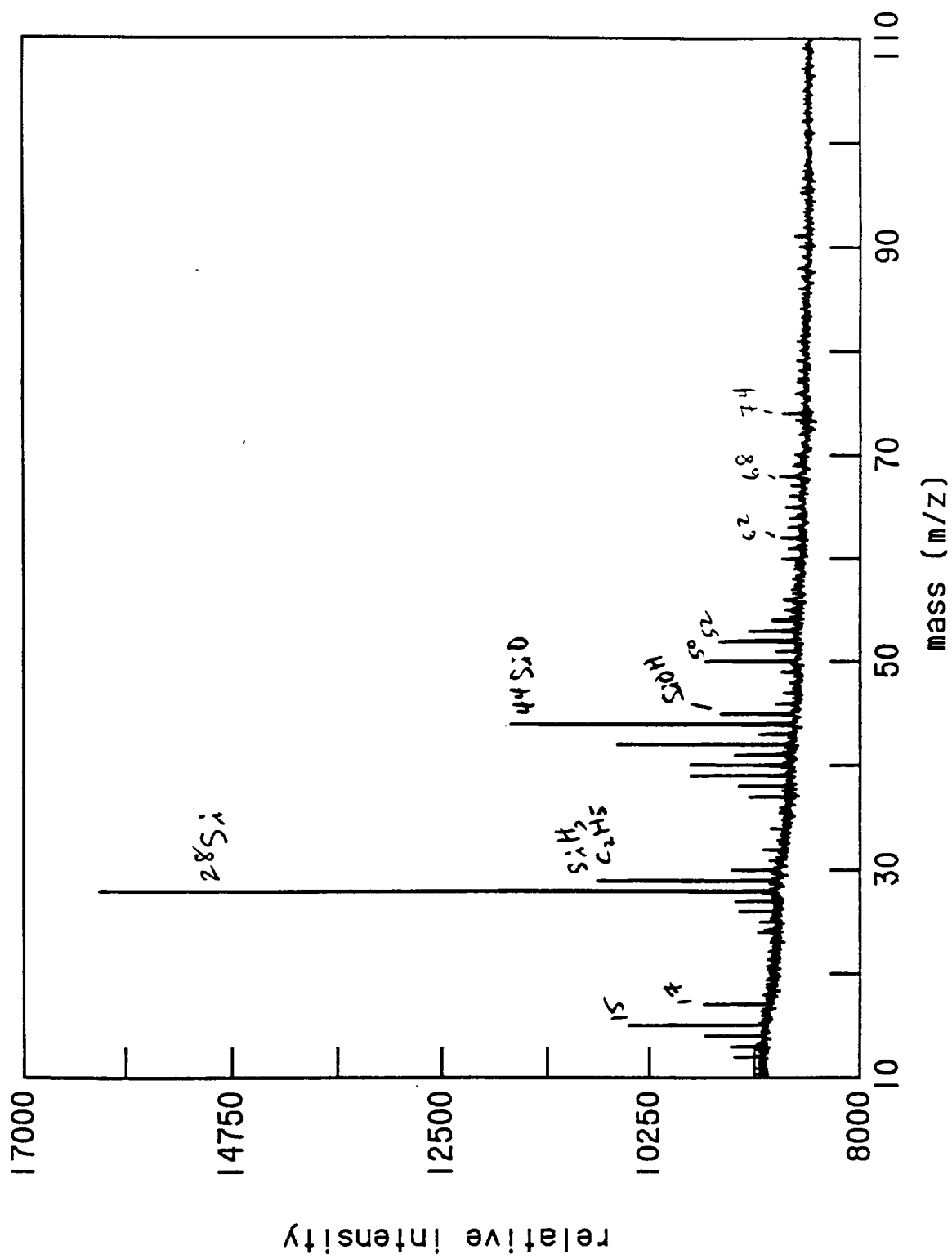
LDEF mirror CM01-52 fe2325

+  $\sim \frac{1}{2}$  ML sputter erosion



LDEF mirror CM01-52 fe2327

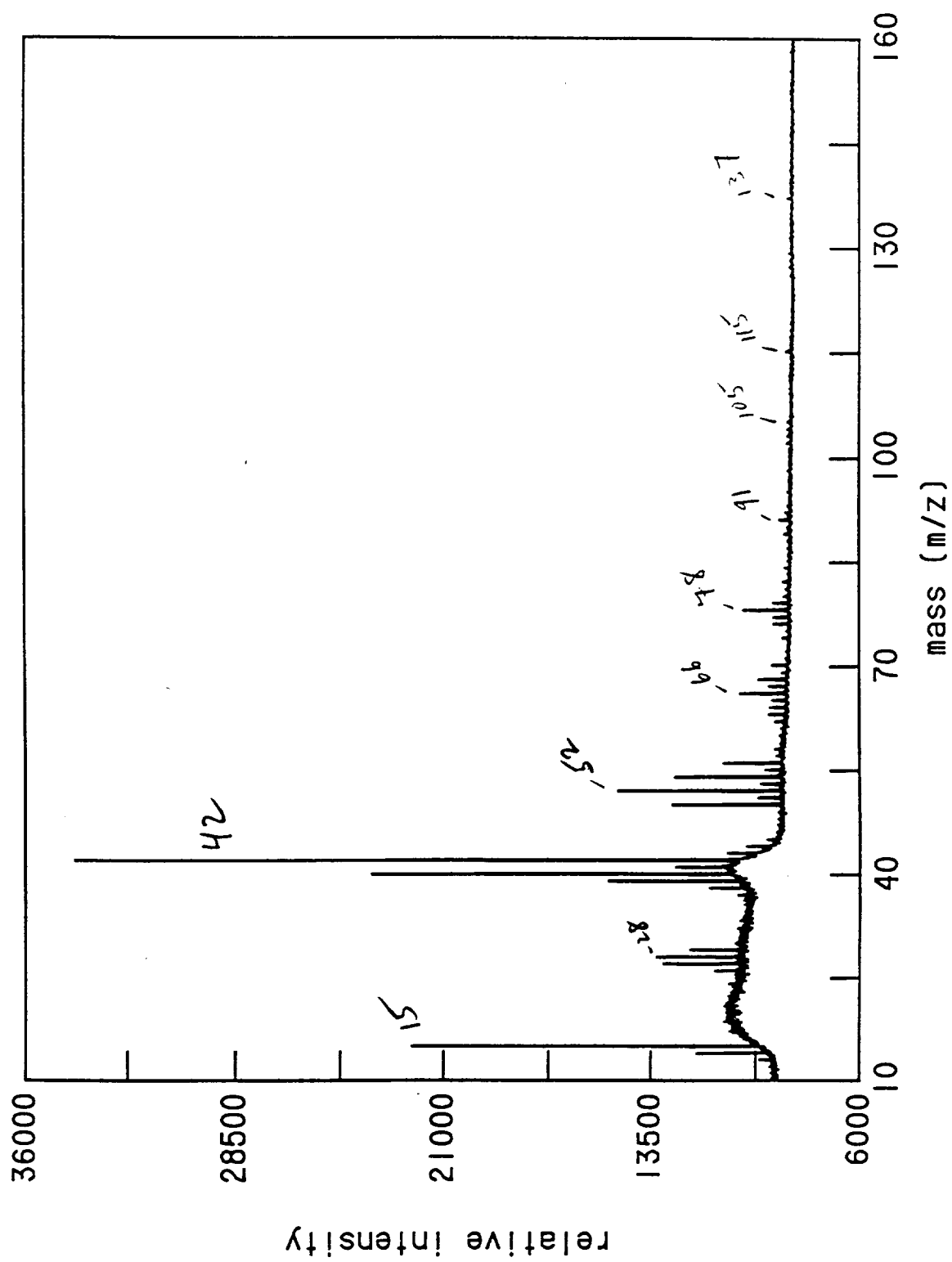
~ 1 ML sputter erosion



LDEF mirror CM01-52 ~~CM01-52~~ fe2323

after ~ 2 ML erosion  
(different spot)



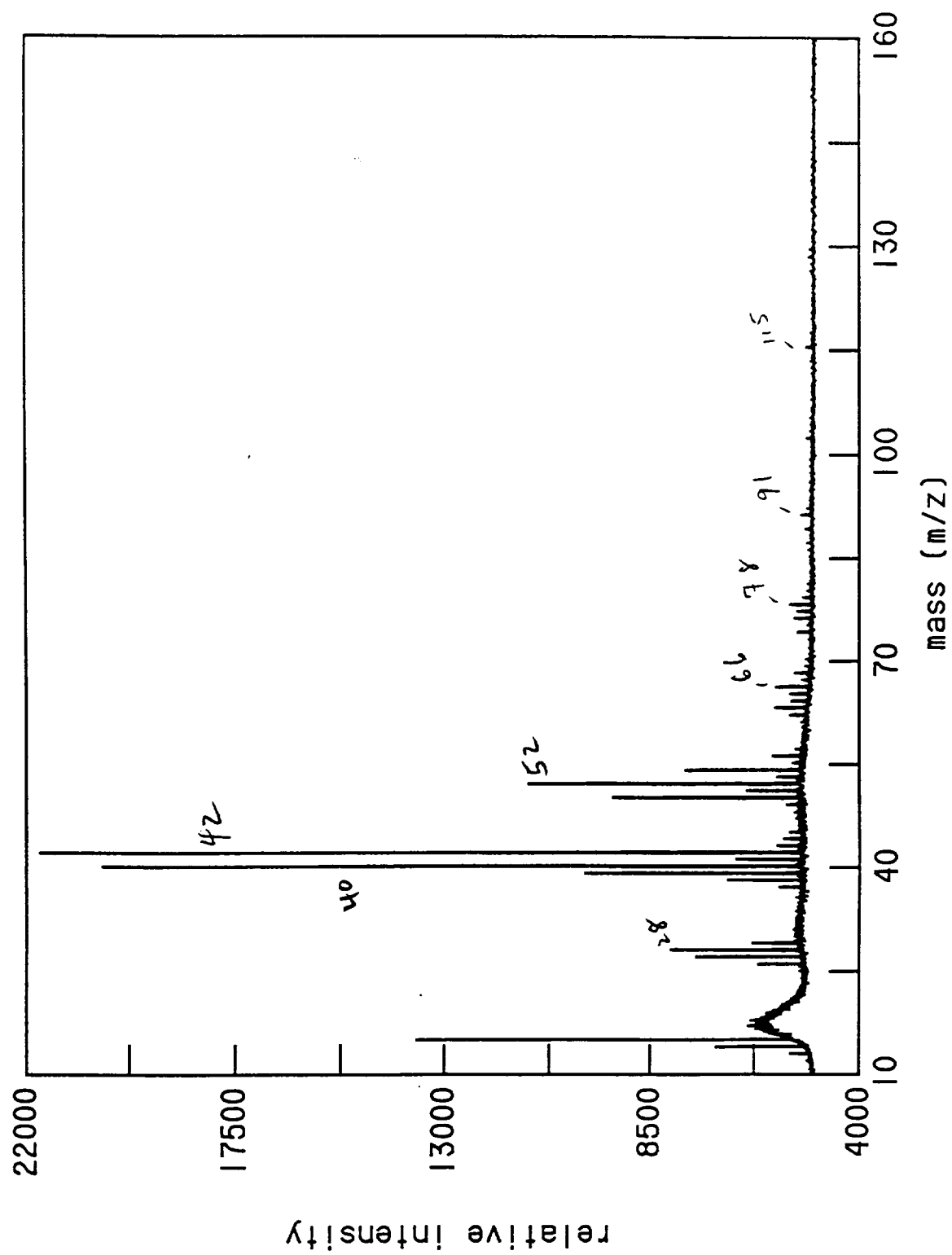


uv-pgl surface fe2317

static conditions

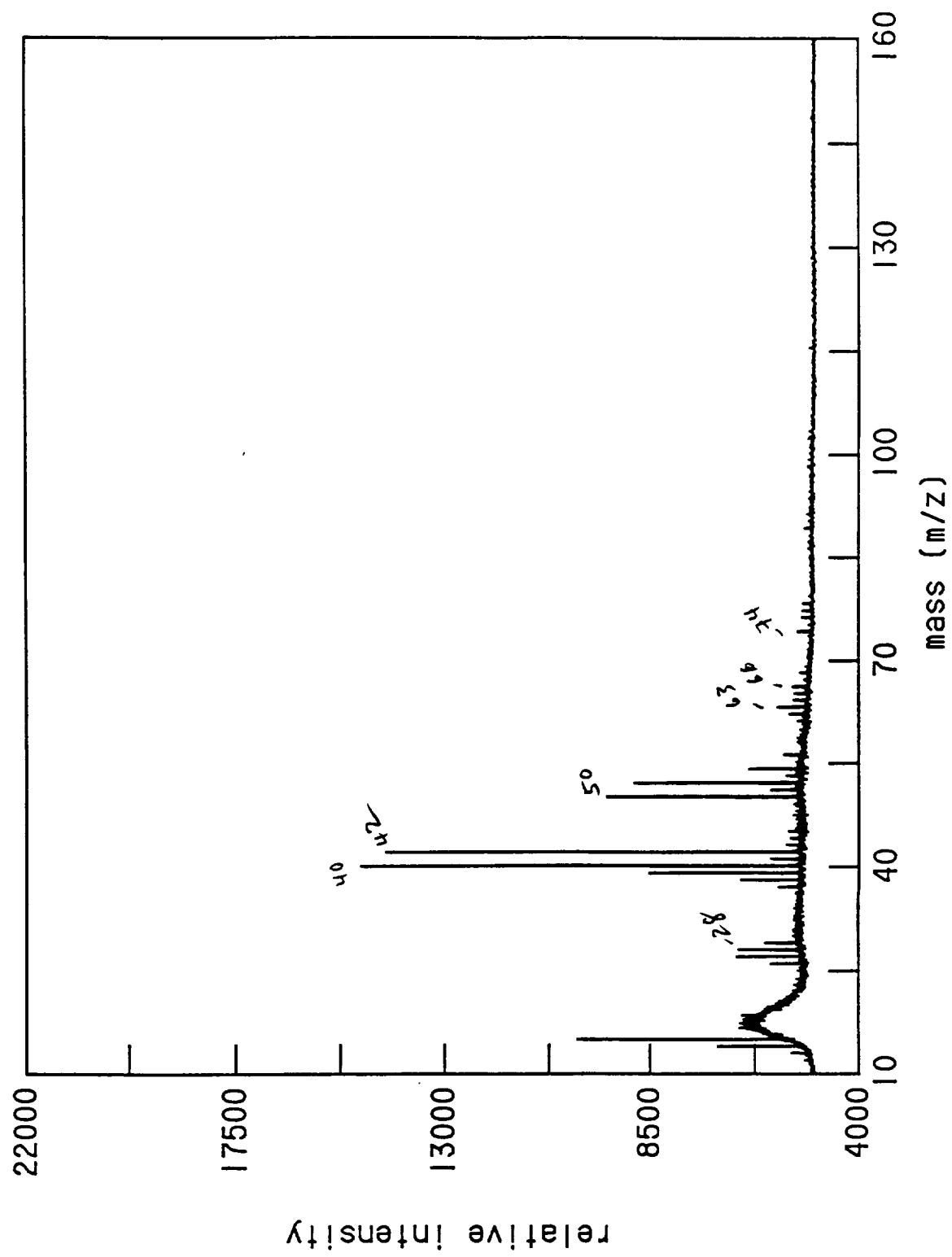
charging: e<sup>-</sup> gun used for this sample

ORIGINAL PAGE IS  
OF POOR QUALITY



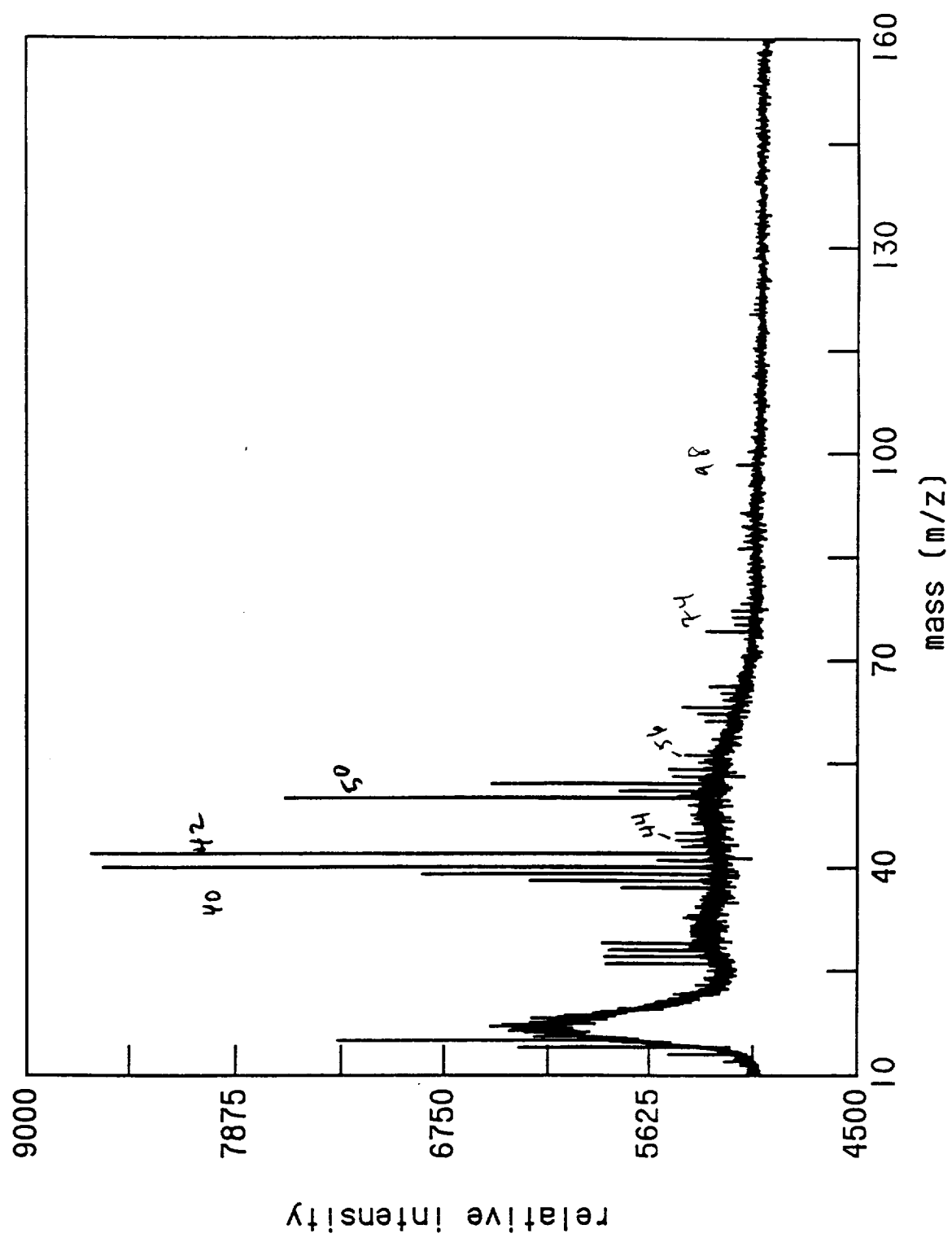
UV-PGI, fe2406 surface

new spot  
static conditions



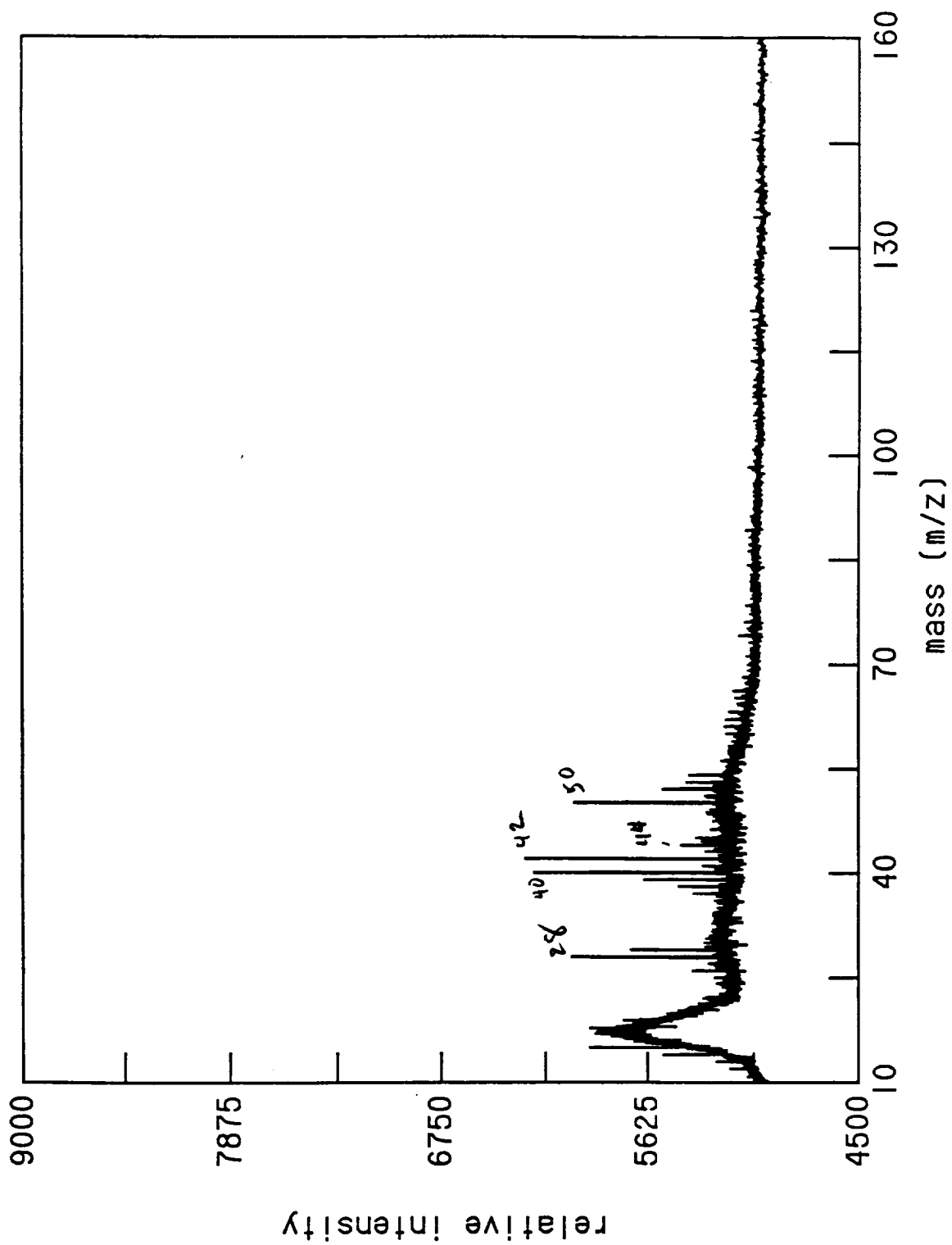
UV-PGI fe2407

$\sim \frac{1}{2}$  ML erosion



UV-PG1 fe2408

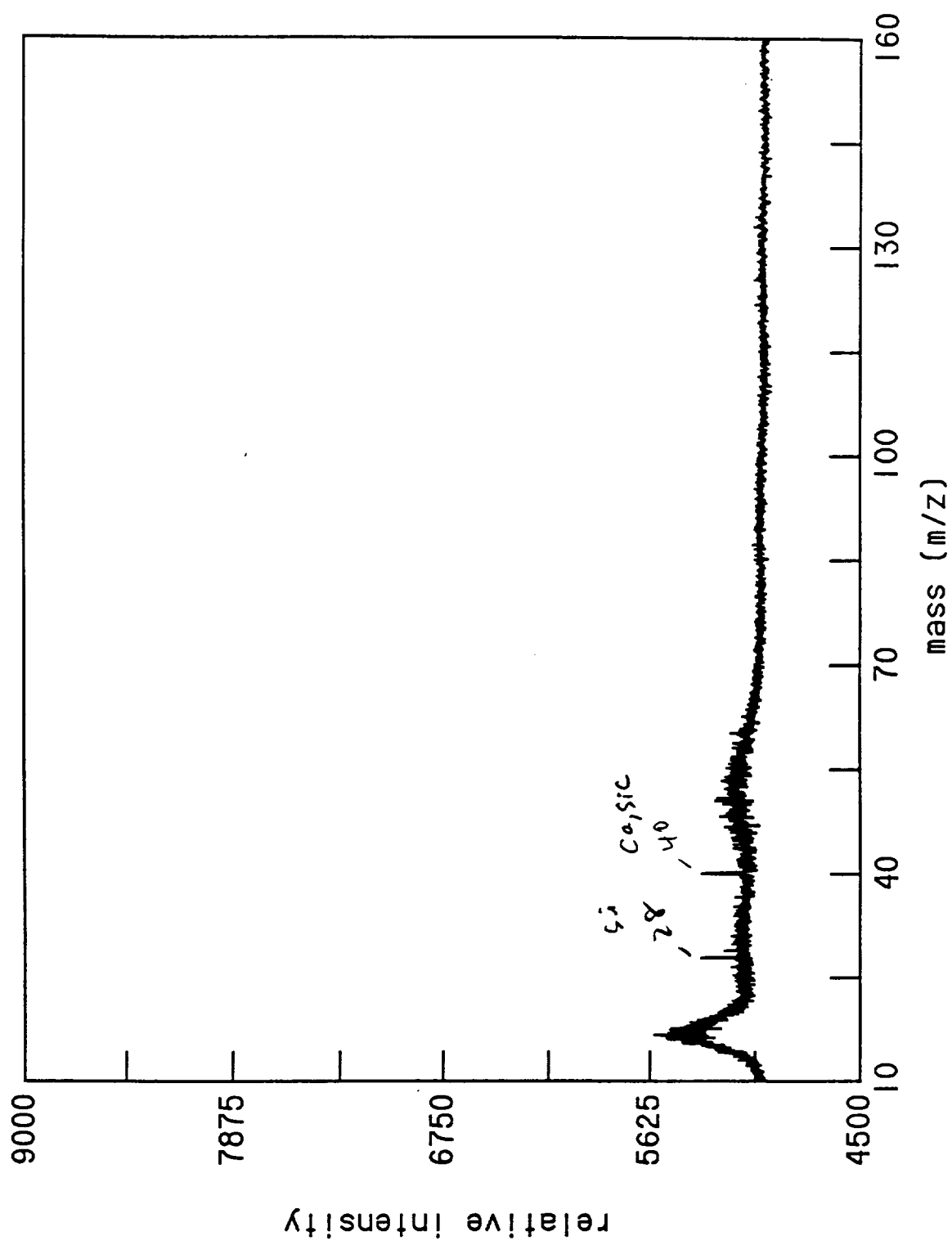
$\sim 1/2$  ML emission



UV-PGI fe2409

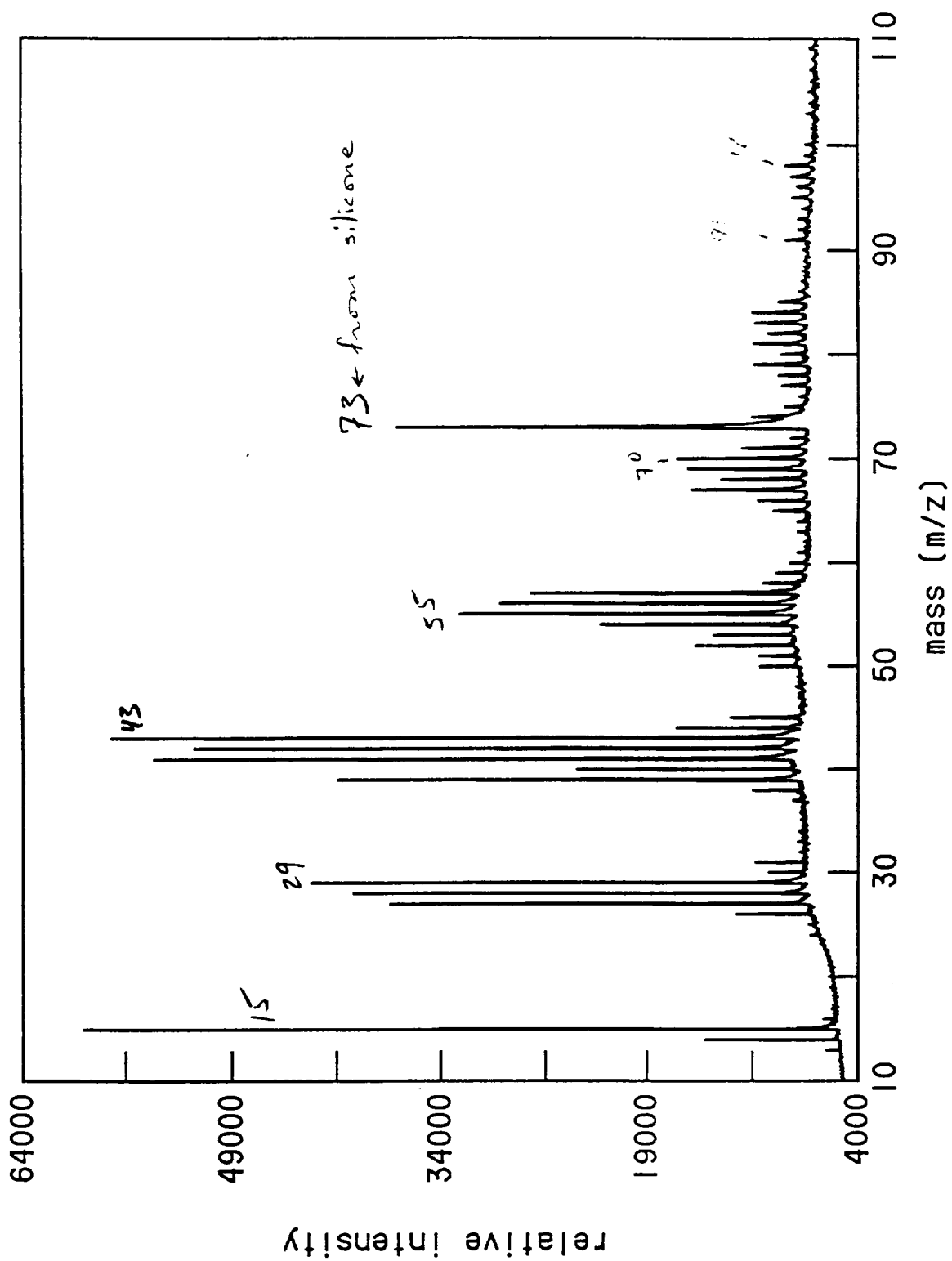
*~4 MZ emission?  
(+ charging)*

ORIGINAL IMAGE IS  
OF POOR QUALITY



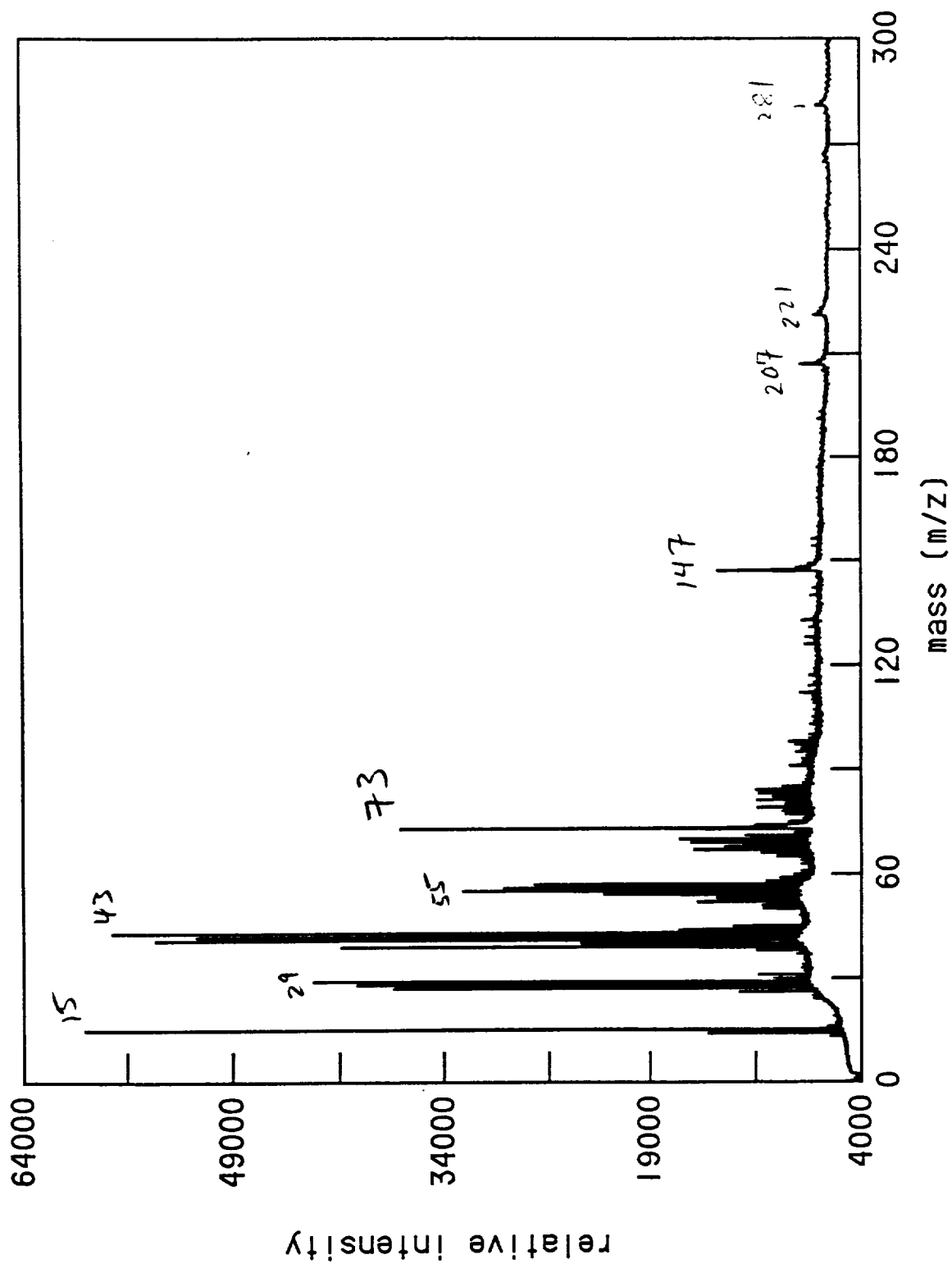
UV-PGI fe2410

~8 ML erosion?  
cf. 11/11/88



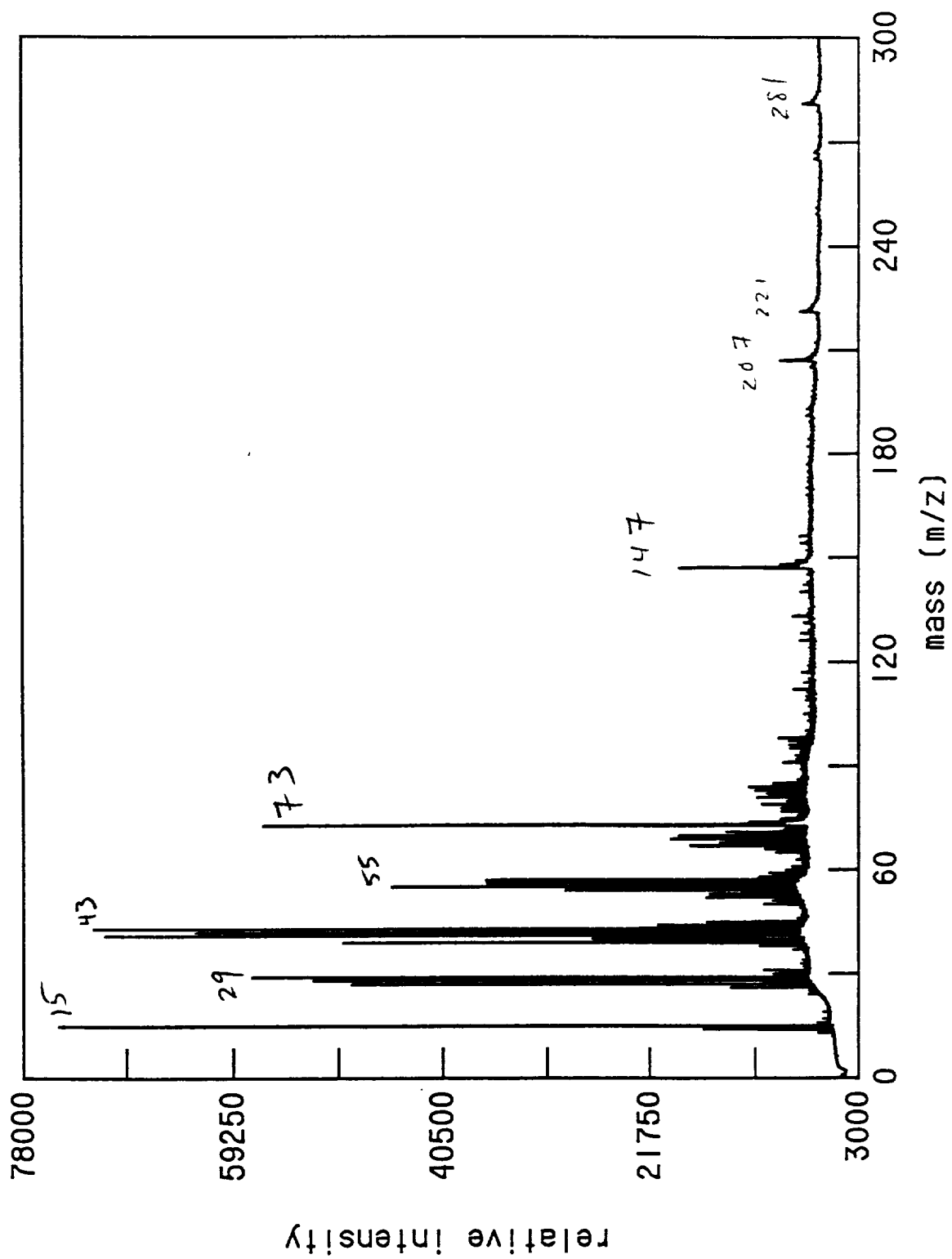
MgF2 mirror 28-92 fe2328 surface  
static conditions

ORIGINAL FILE IS  
OF POOR QUALITY



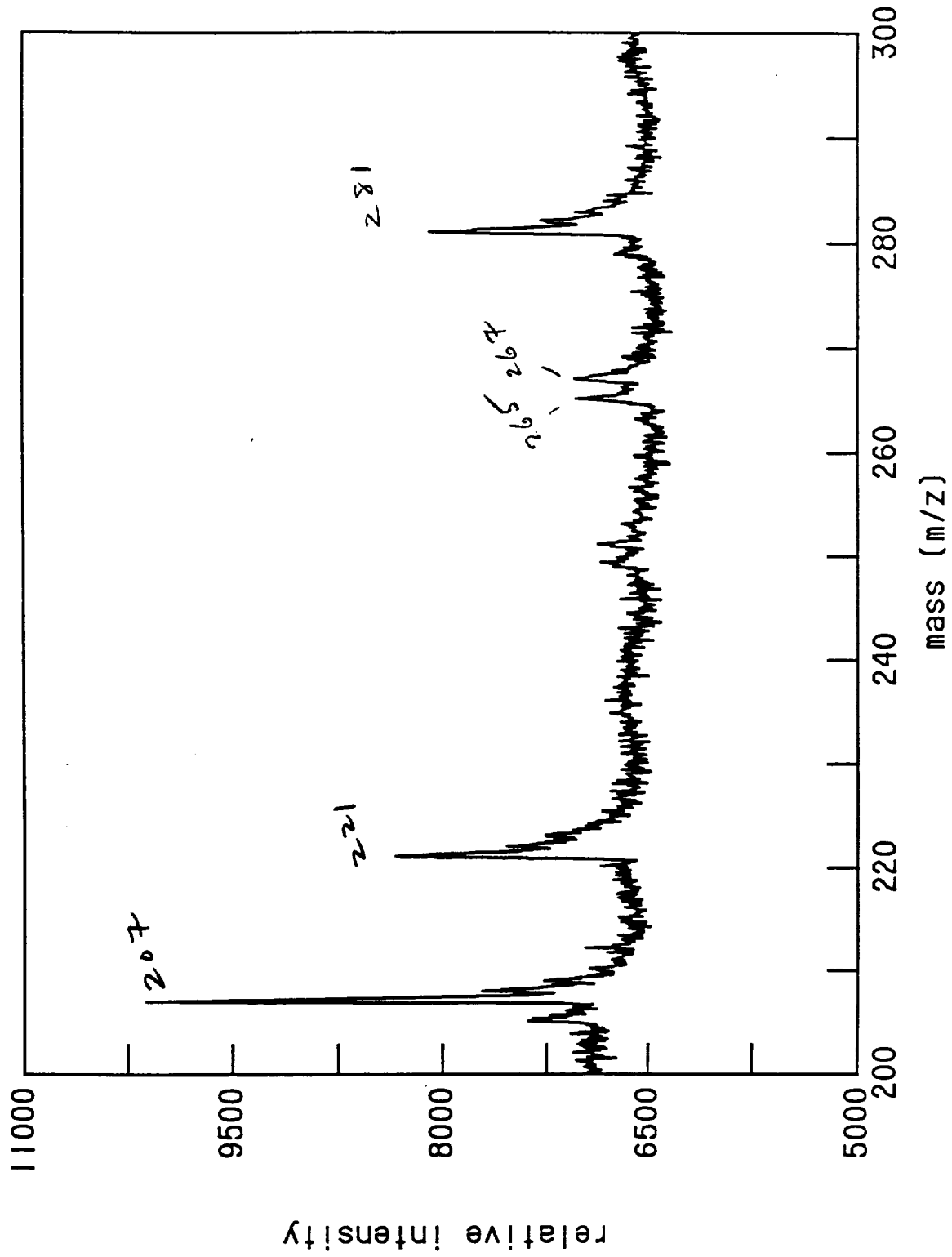
MgF2 mirror 28-92 fe2328 surface  
major silicone peaks: 73, 147, 207, 221, 281





mgF2 mirror 28-92 fe2333

new spot, surface, static conditions

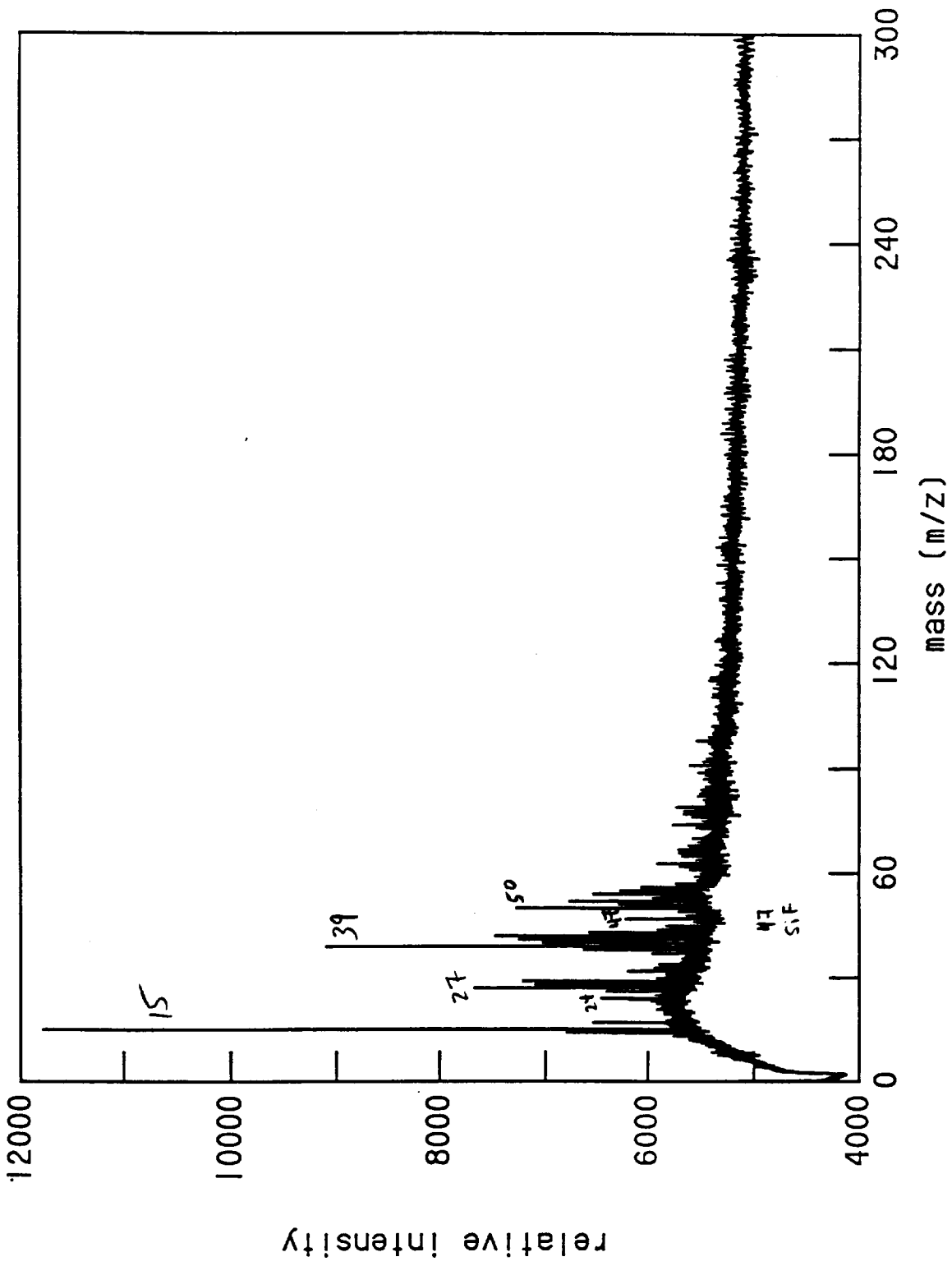


fe2333 2/23/93 MgF2 mirror

siloxane peaks

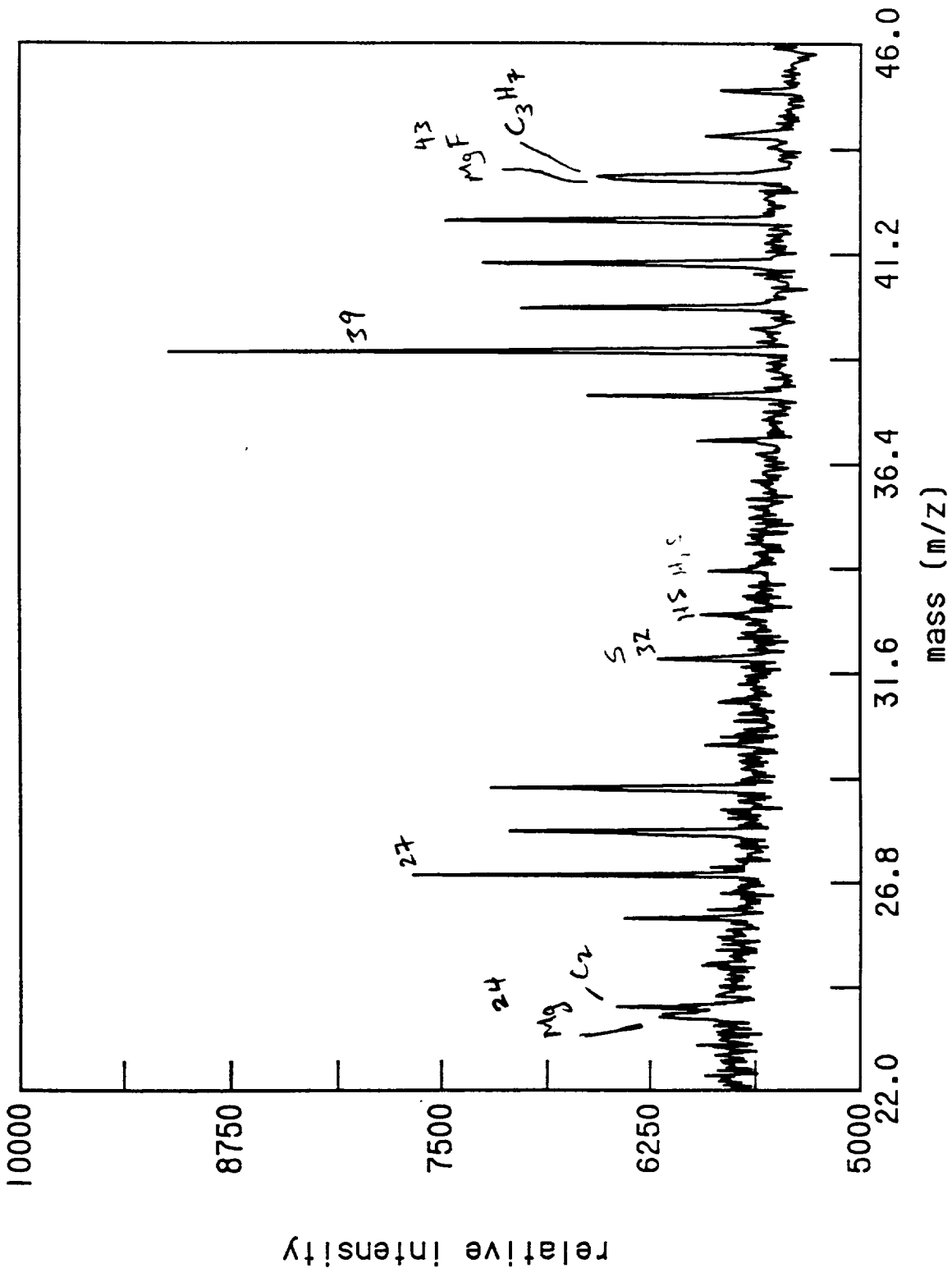
between 200 and 300 amu.

(peaks seen to ~350 amu)



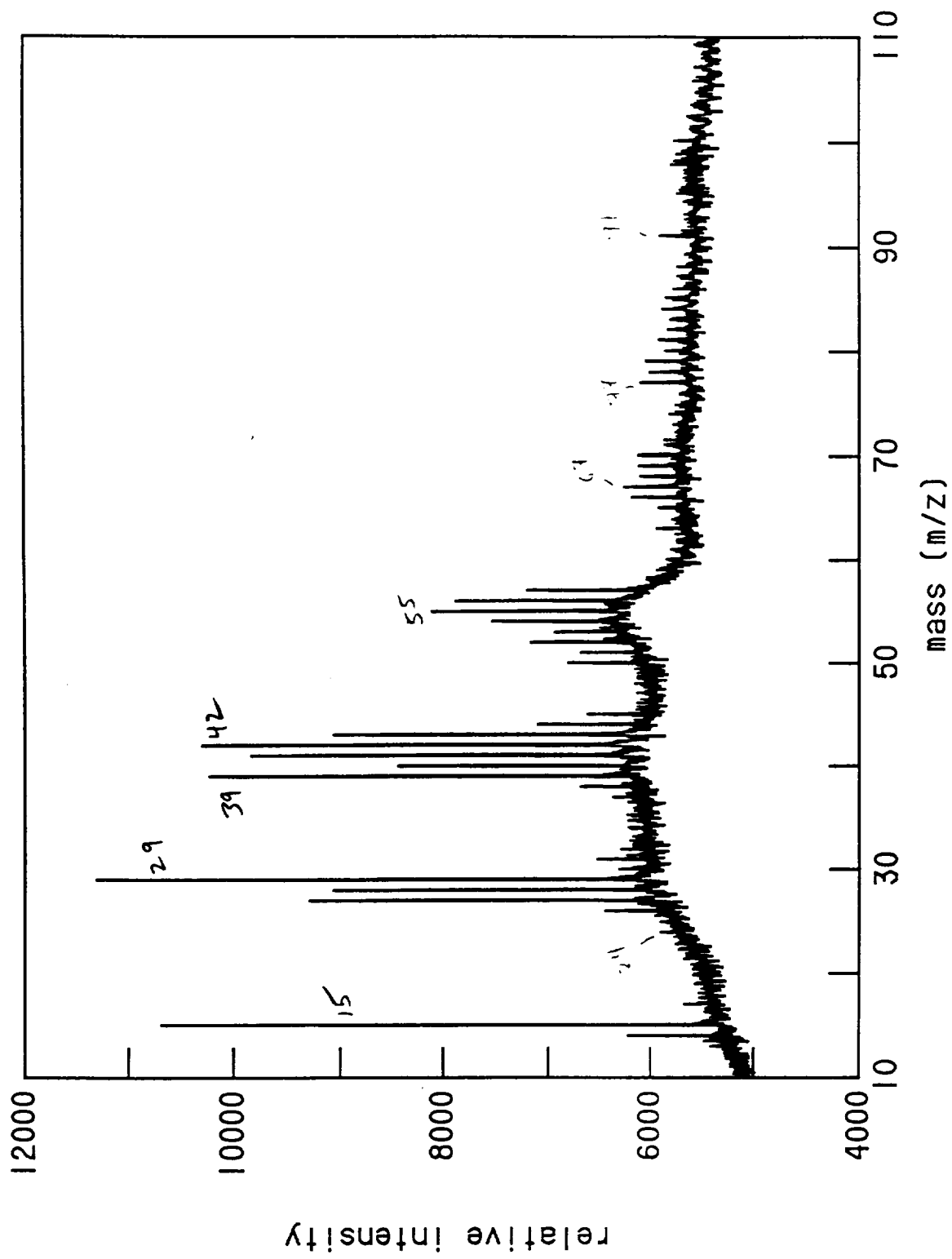
MgF2 mirror 28-92 fe2334

+ ~ 1 ML erosion

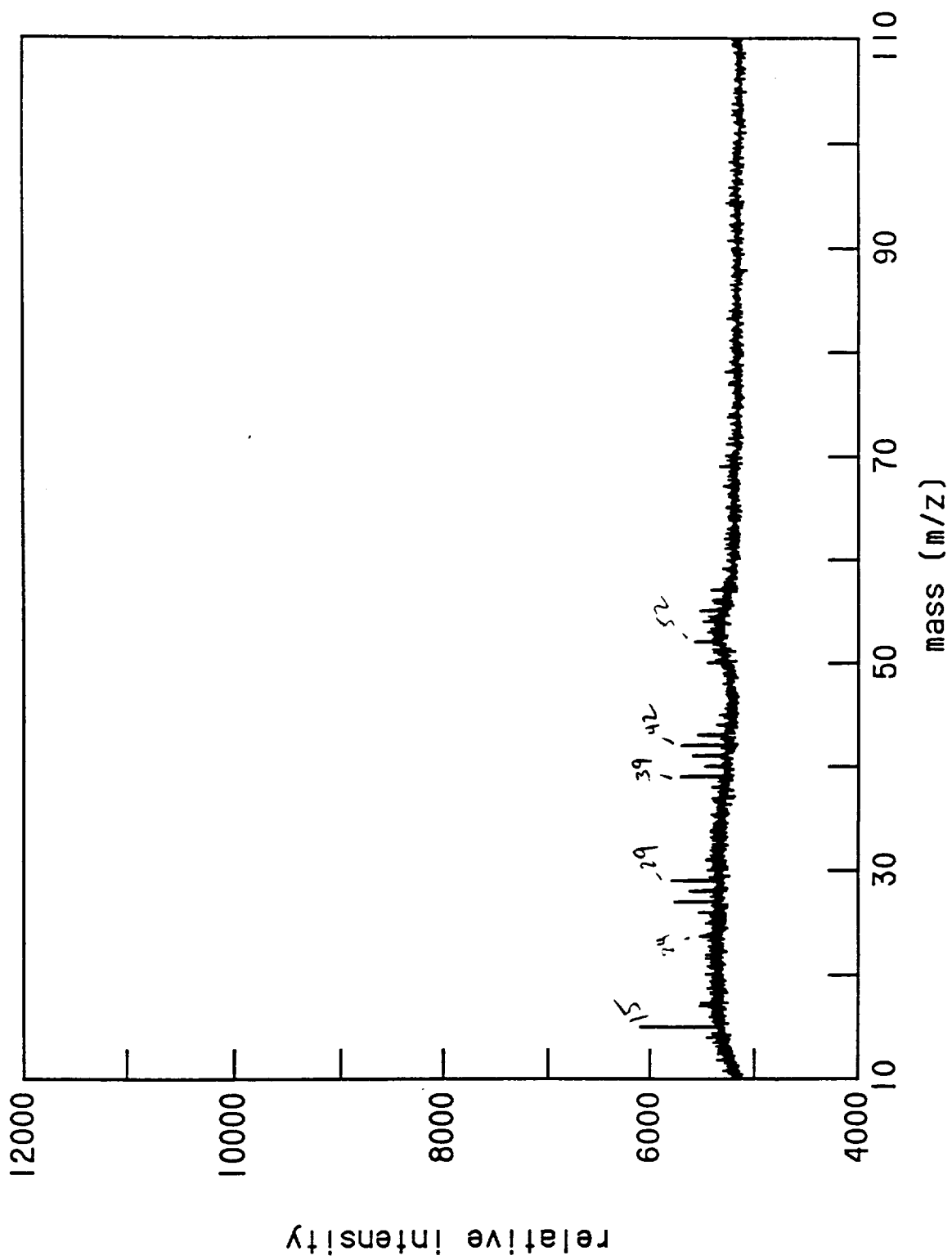


MgF2 mirror 28-92 fe2334

enlarged view



MgF2 mirror SI control fe2337 surface  
*static conditions*

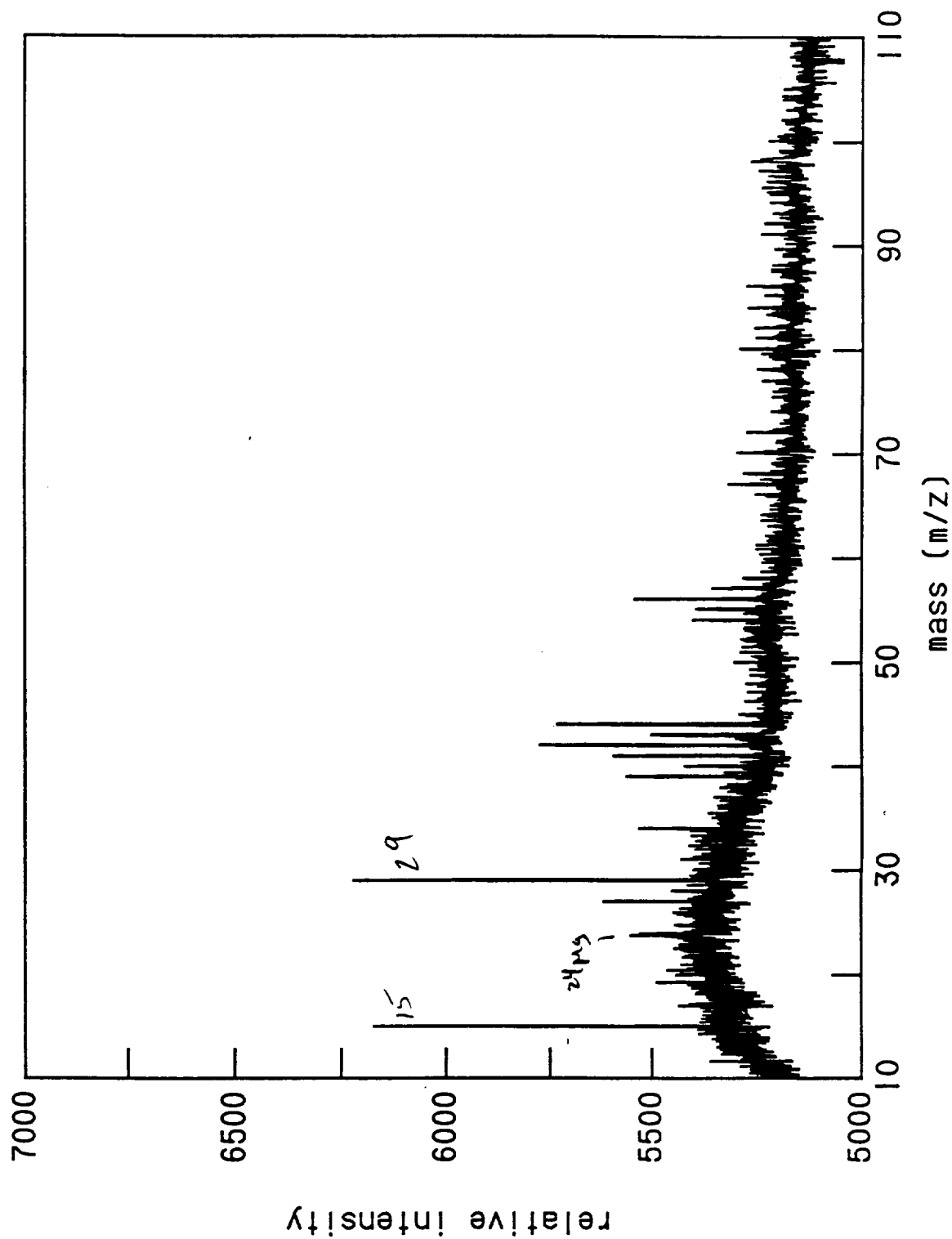


MgF2 mirror Si control fe2338

+ ~ 1 ML erosion  
1.1.19.19

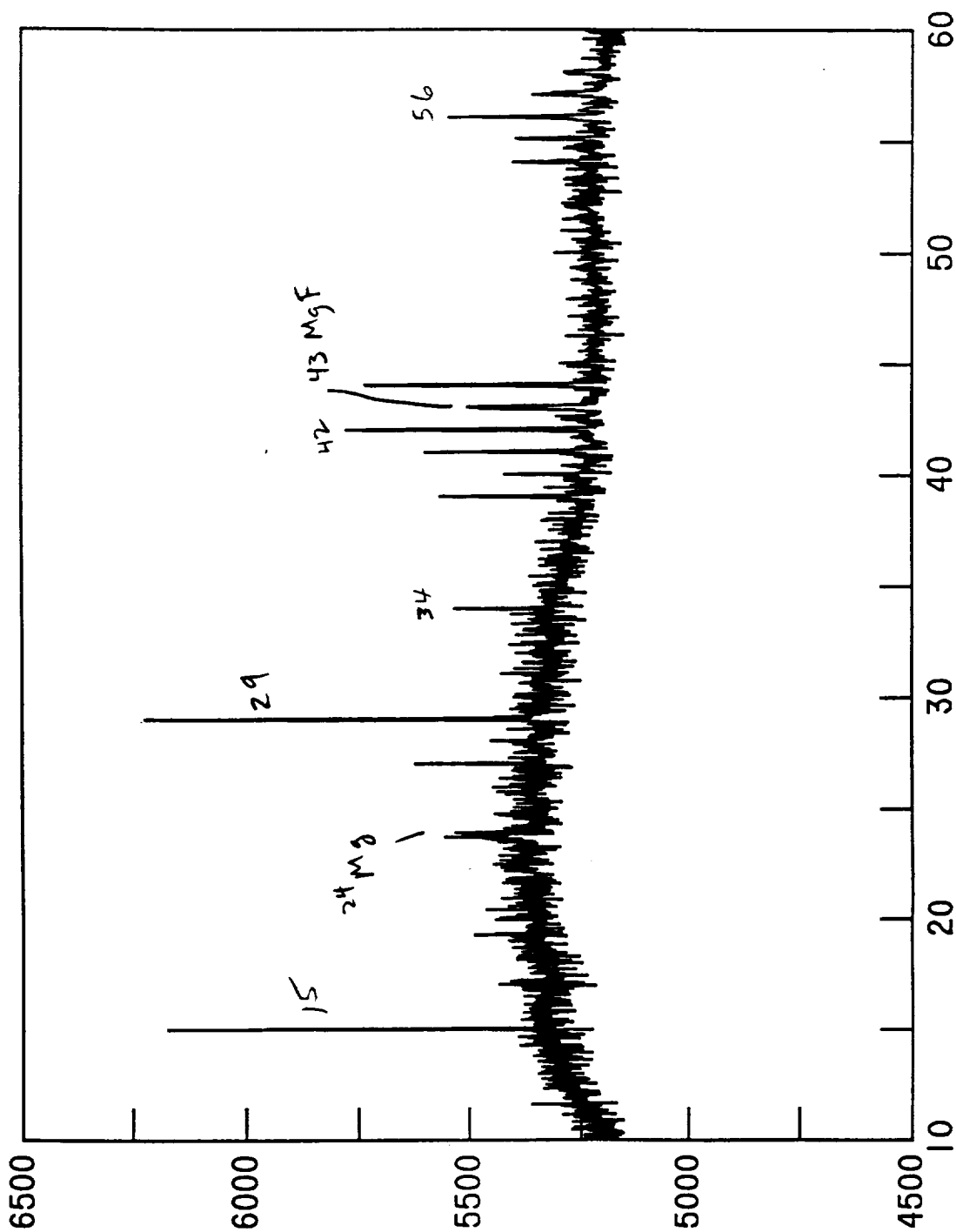
ORIGINAL PAGE IS  
OF POOR QUALITY

ORIGINAL FILE IS  
OF POOR QUALITY



MgF2 mirror SI control fe2339

- see next figure,  
expanded scale -  
+ 1/2 PL erosion  
+ 1/2 beam



FE2339

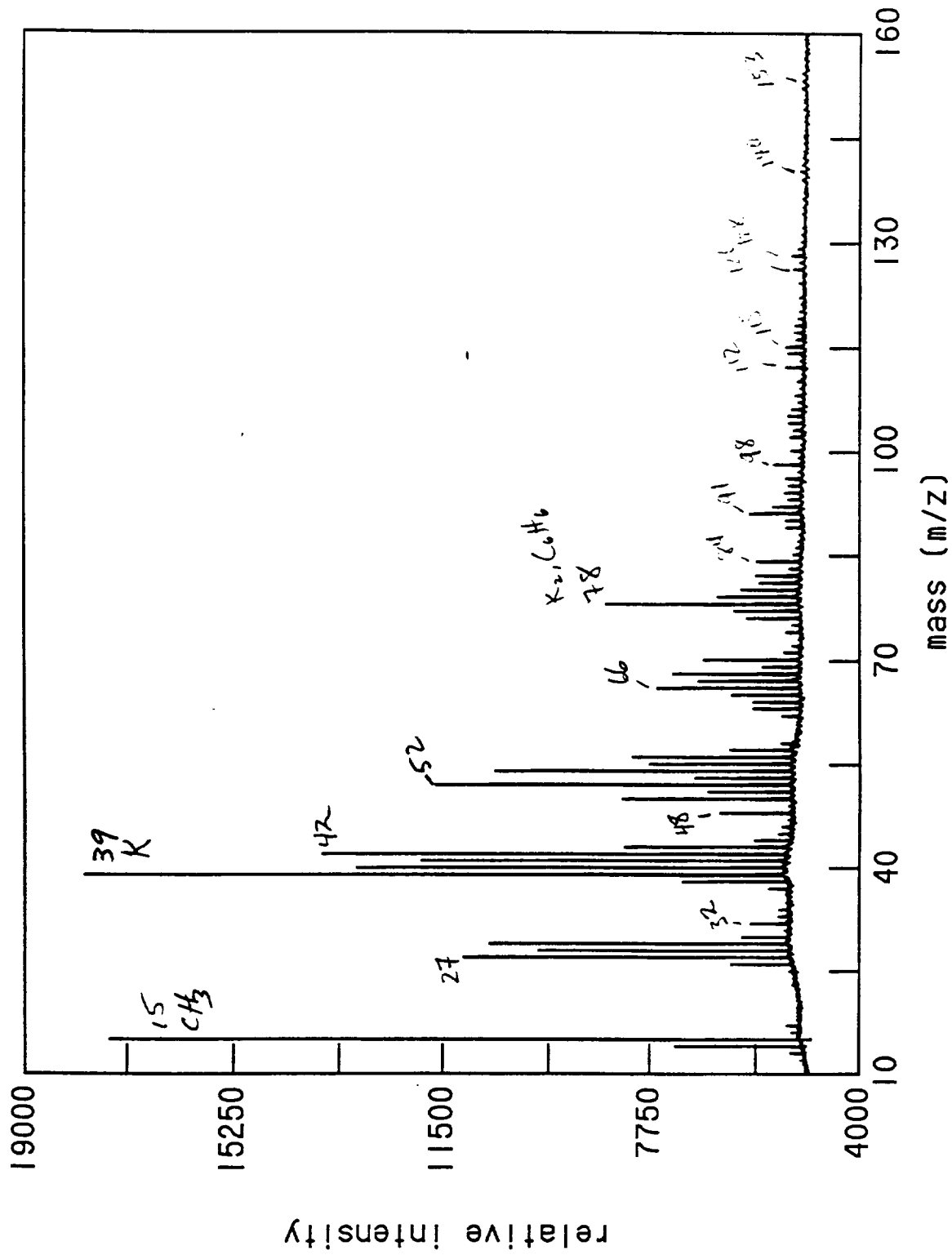
27-FEB-93 14:34:46

MgF<sub>2</sub> mirror  
SI control

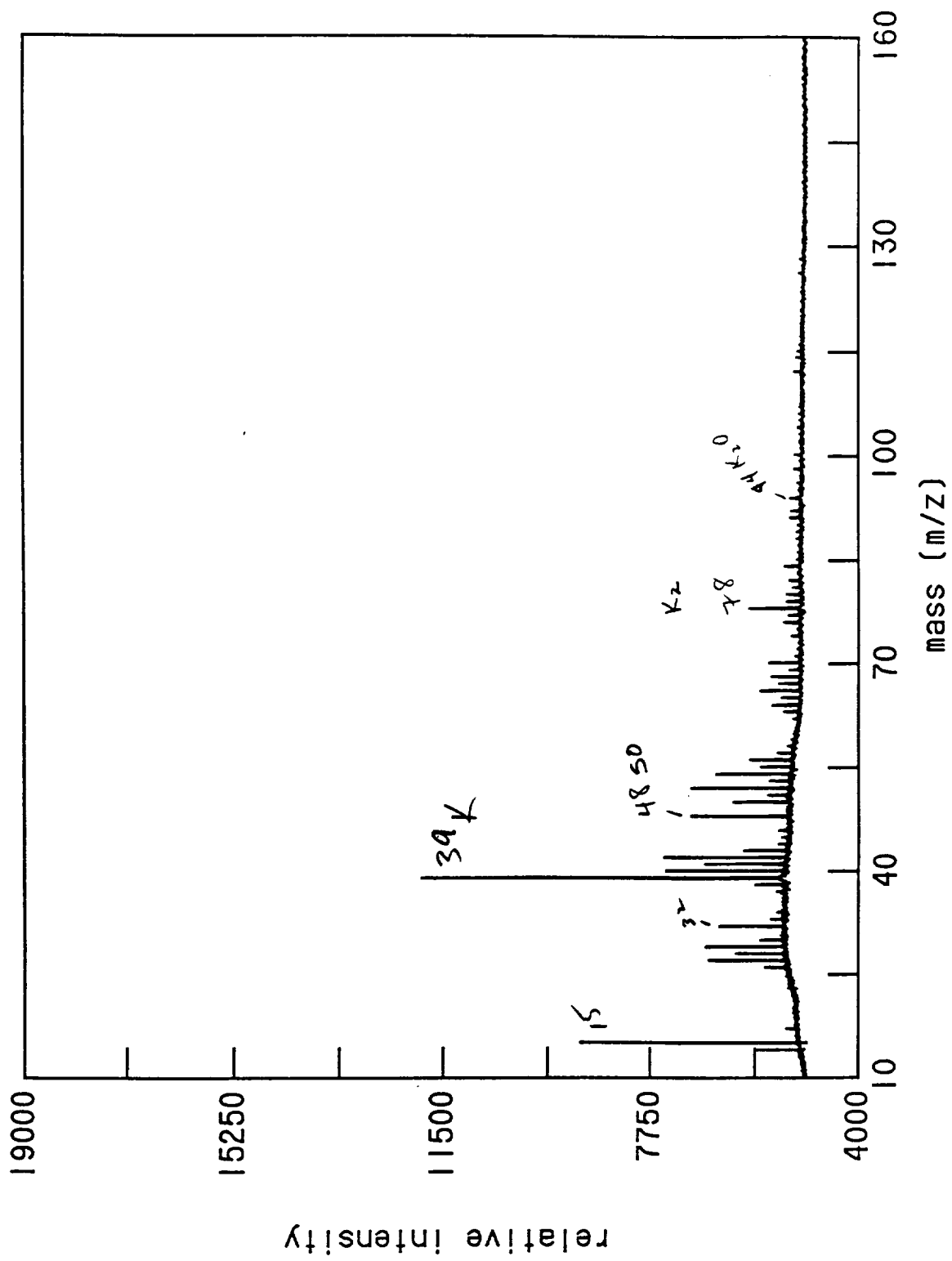
te<sup>-</sup> to mirror



ORIGINAL PAGE IS  
OF POOR QUALITY

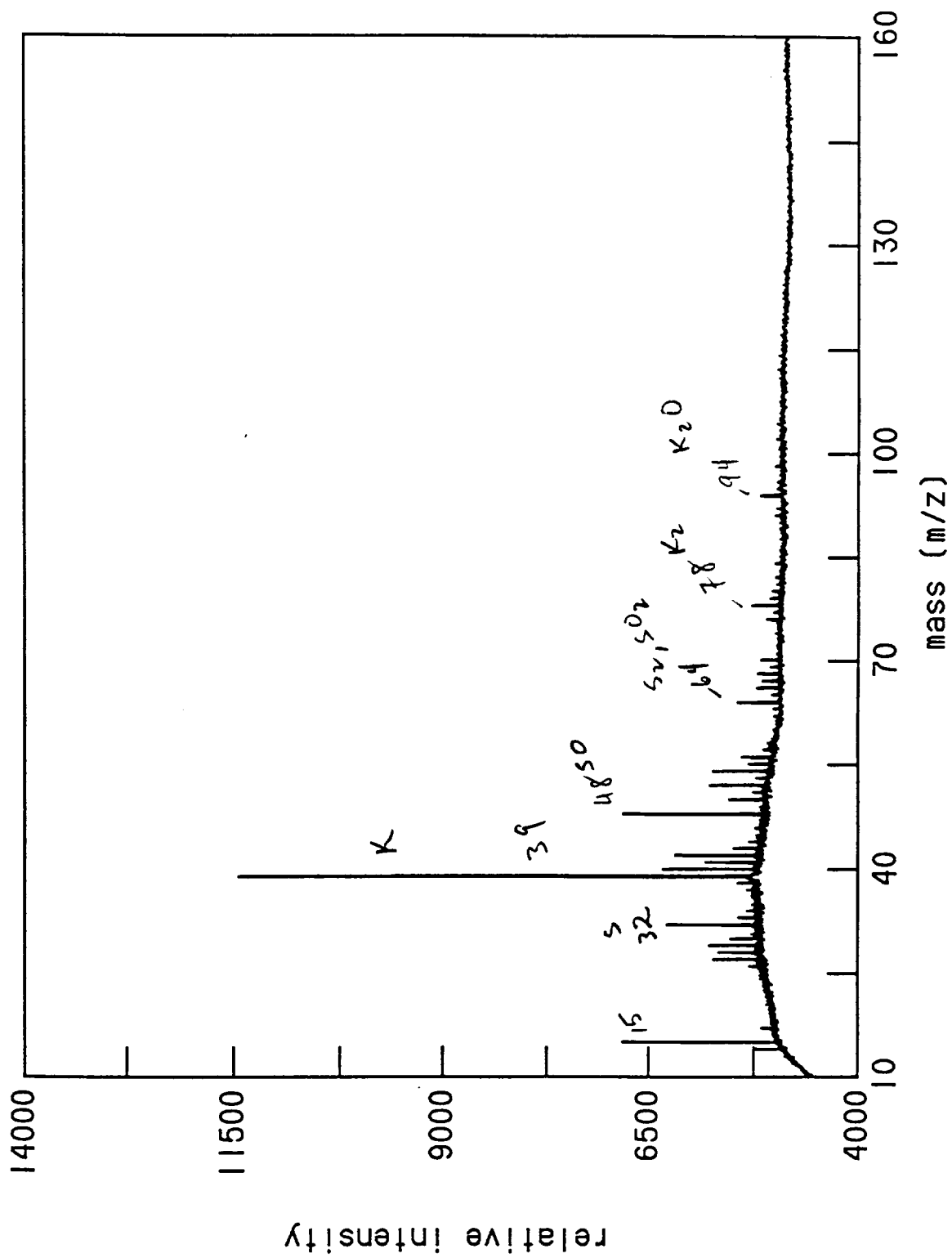


YB-1 LDEF 02-35 surface FE 2412  
static conditions



YB-1 LDEF 02-35 fe2413

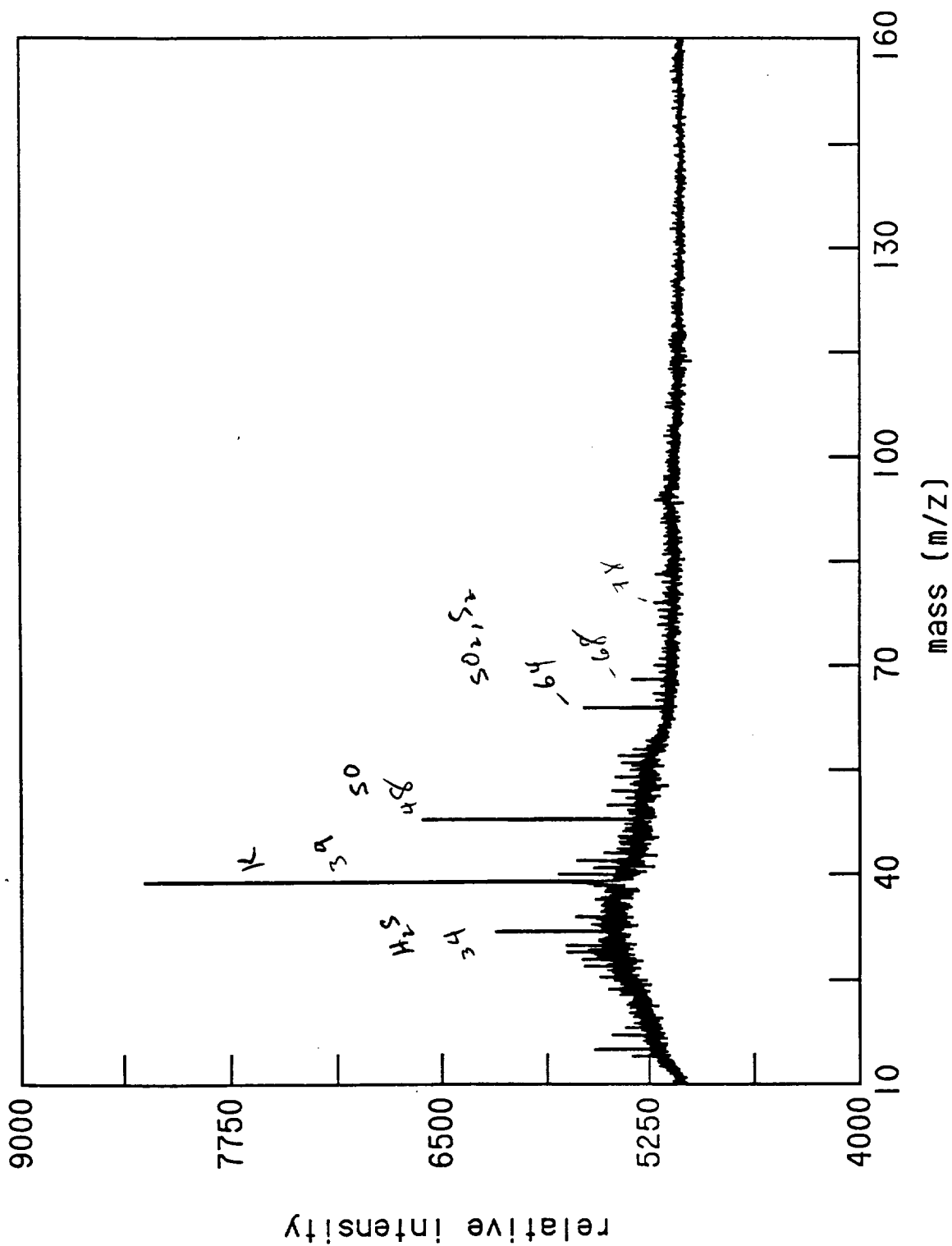
$\sim \frac{1}{2}$  ML erosion



YB-1 LDEF 02-35 fe2415

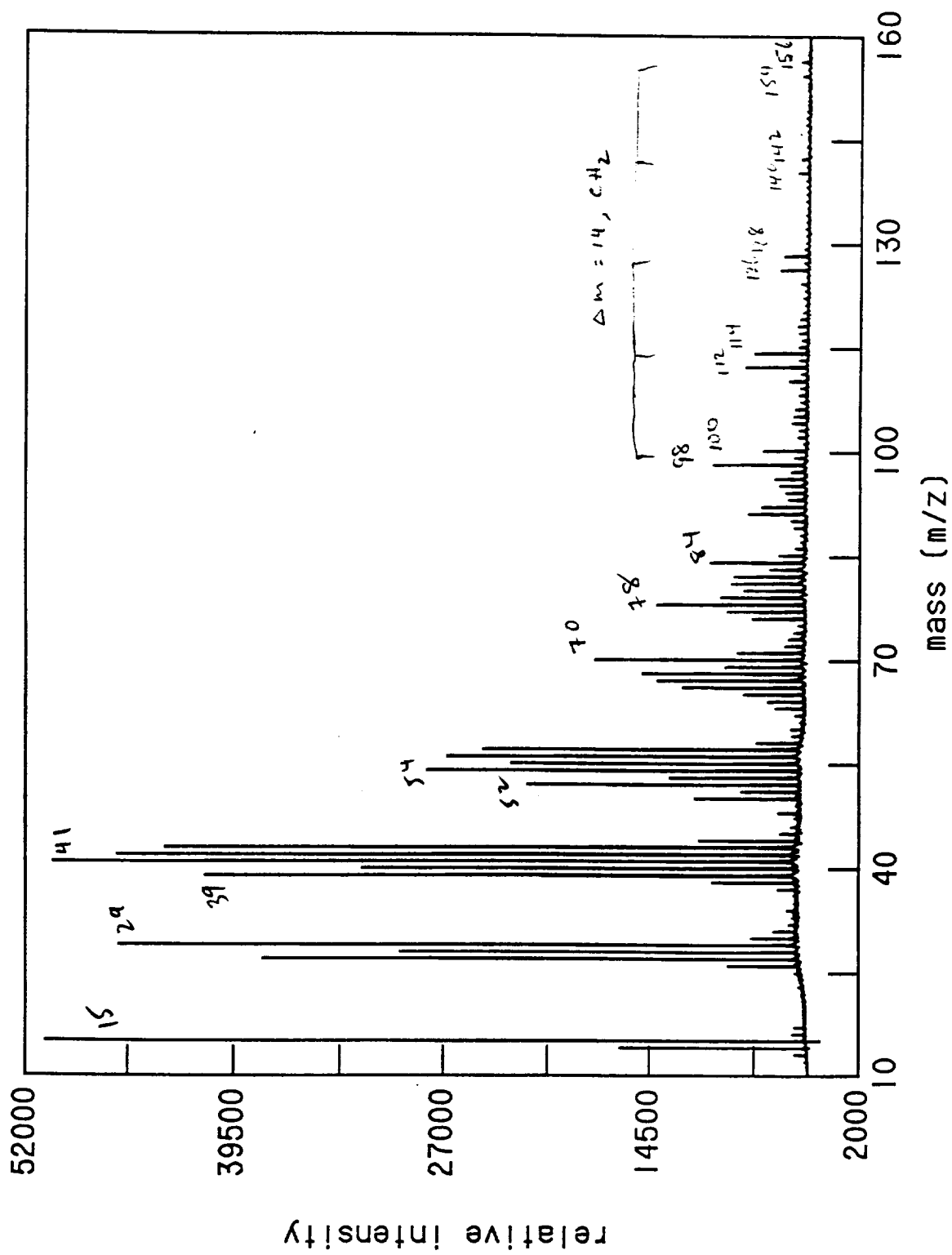
$\sim 2 \frac{1}{2}$  ML erosion

ORIGINAL PAGE IS  
OF POOR QUALITY



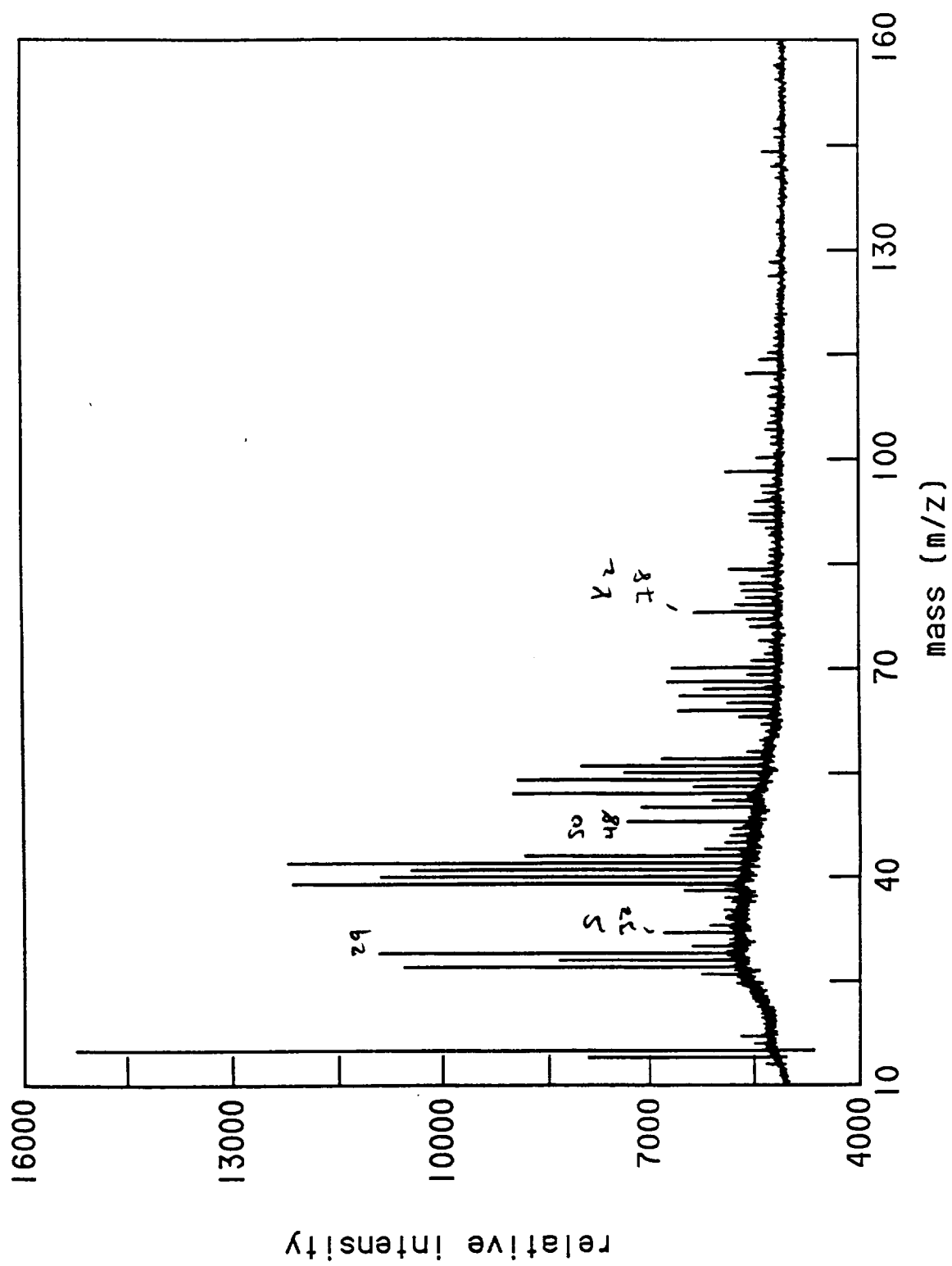
YB-1 LDEF 02-35 fe2419

more sputtering  
+ some charging



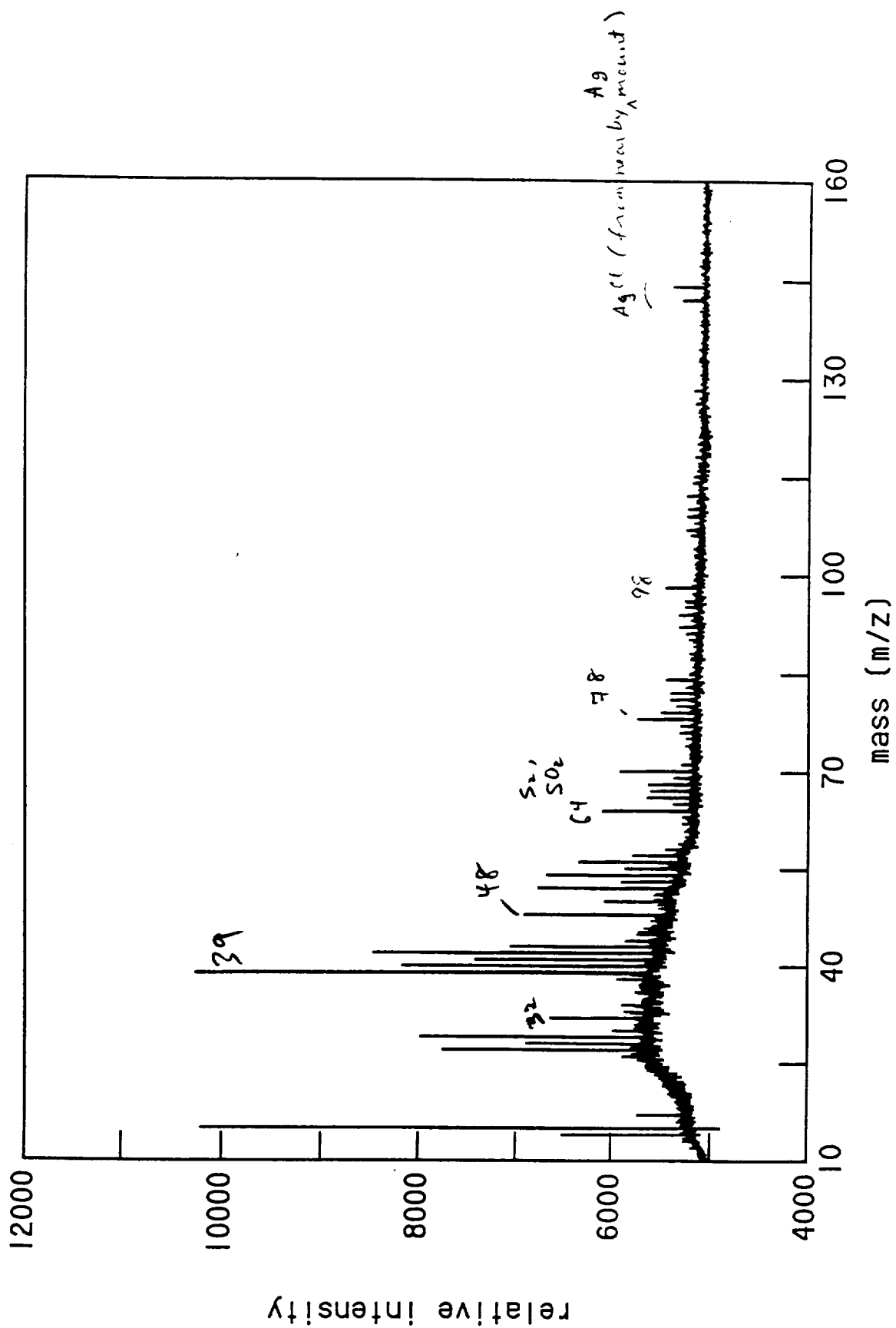
YB-1 control fe2420 surface

*static conditions*



YB-1 control fe2421

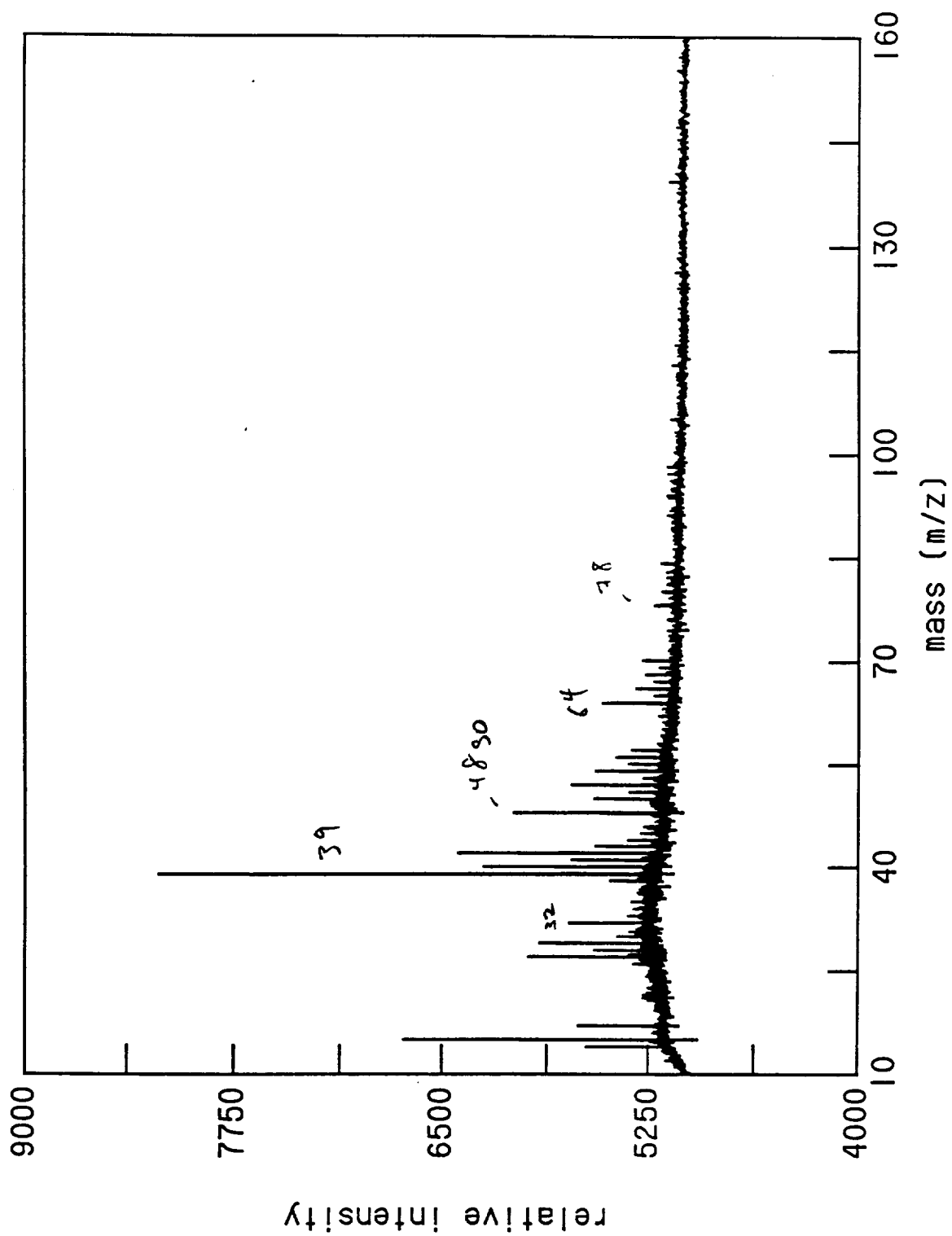
$\sim \frac{1}{2}$  ML excision



YB-1 control fe2423

~2 ML erosion

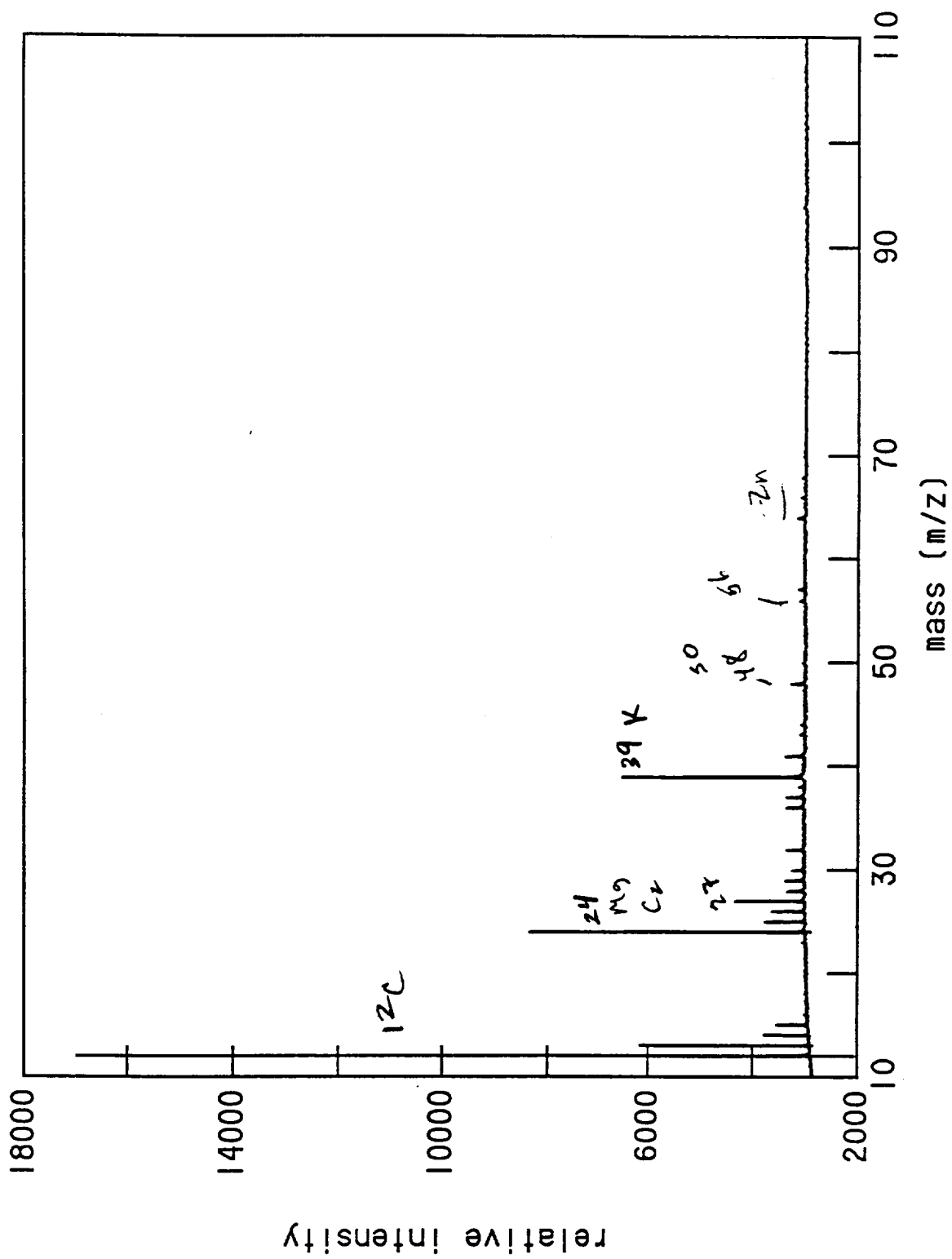
ORIGINAL PAGE IS  
OF POOR QUALITY



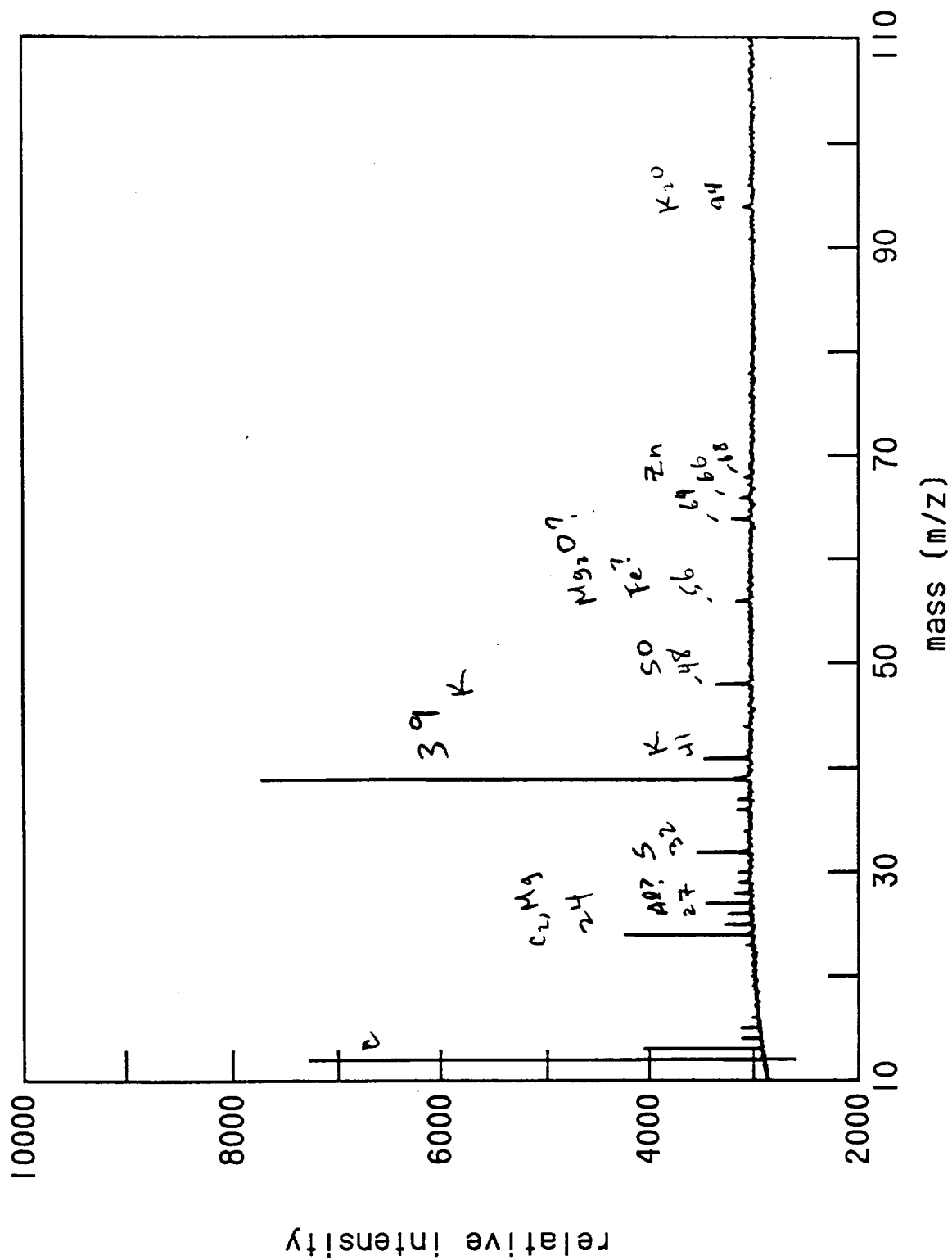
YB-1 control fe2427

new spot  
~ 5 ML evasion?  
1.500 g/100g



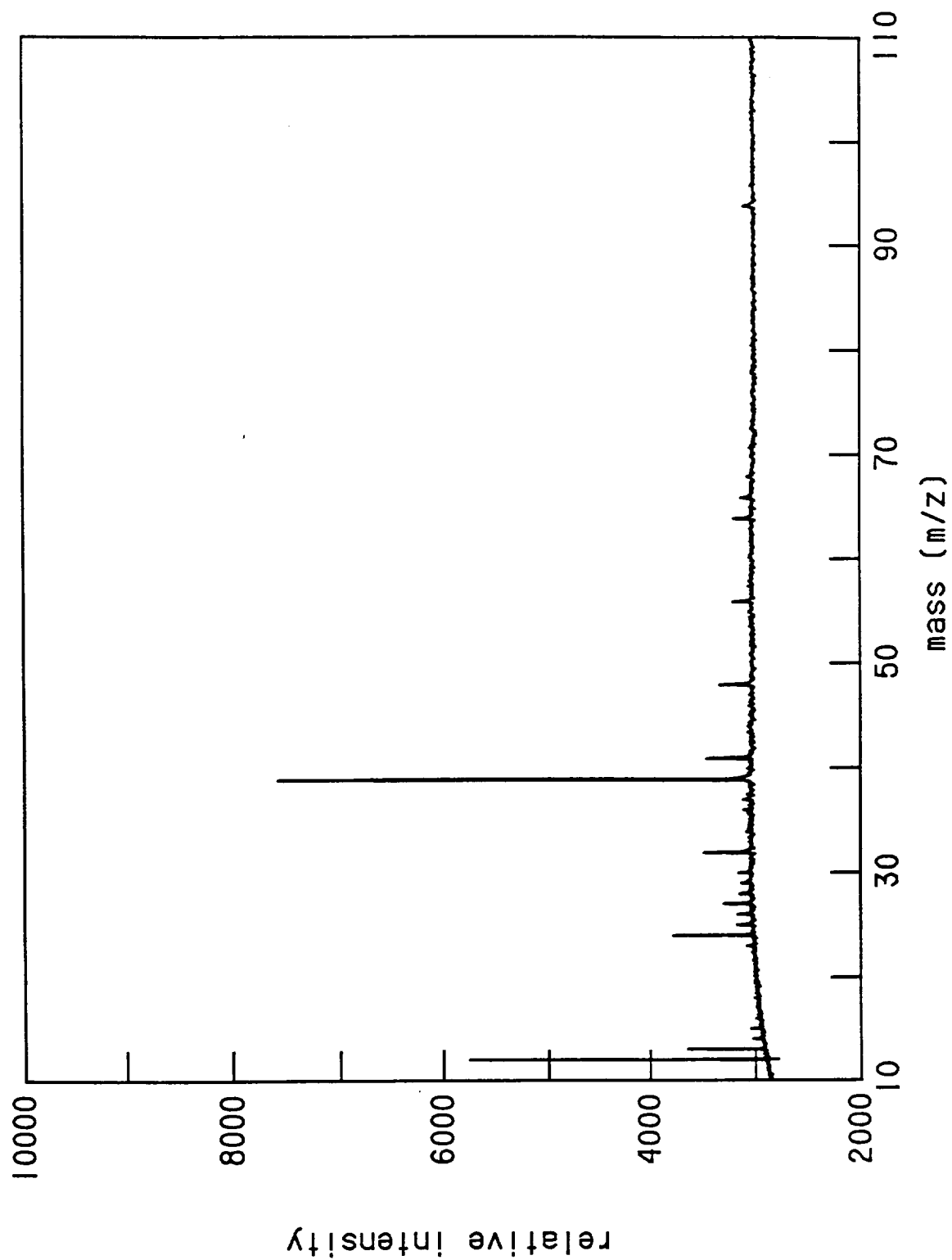


YB-1 LDEF 02-35 surface fe2505 268nm  
static conditions



YB-1 LDEF 02-35 fe2506 266 nm

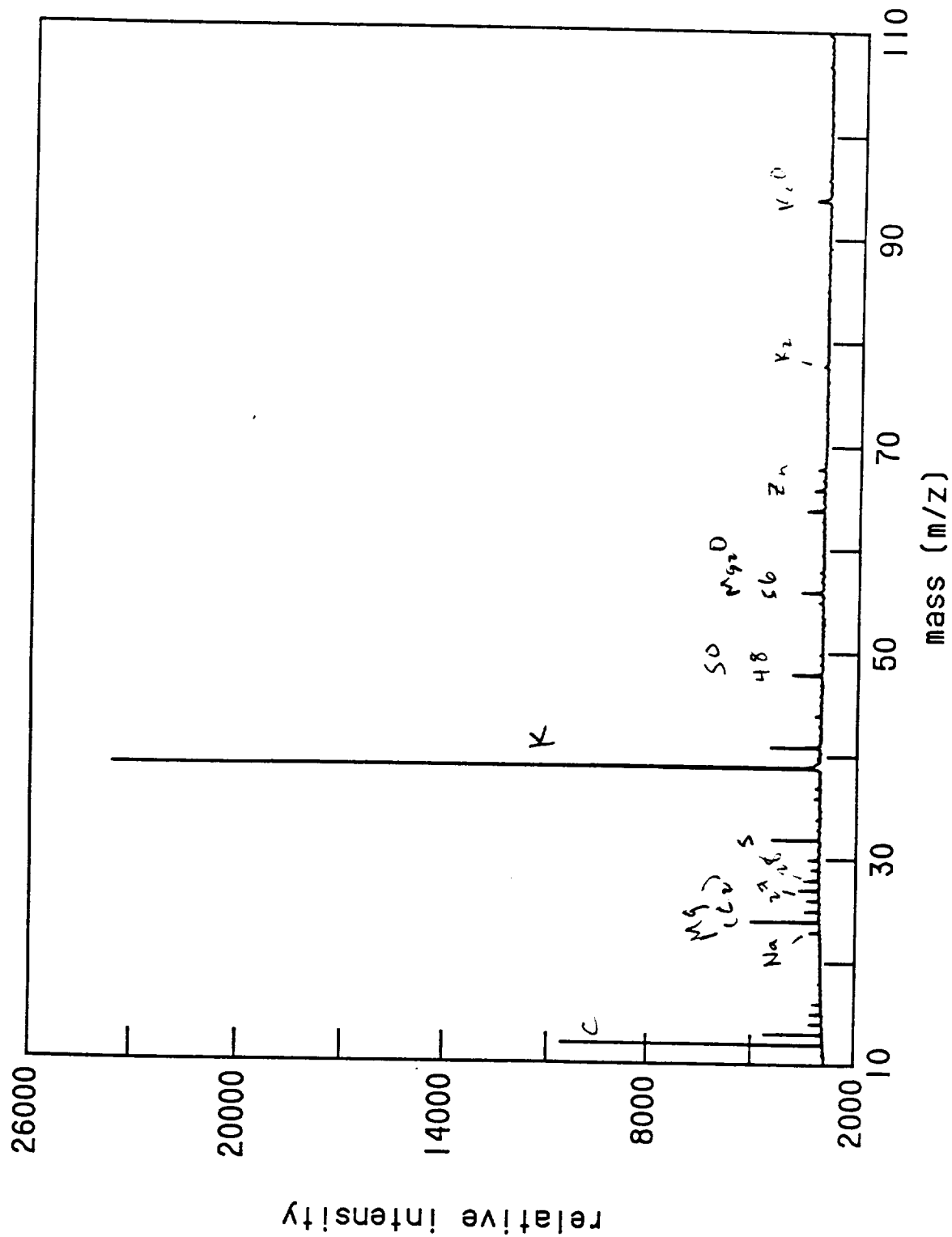
$\sim \frac{1}{2}$  ML emission



YB-1 LDEF 02-35 fe2507 266 nm

a 1ML emission

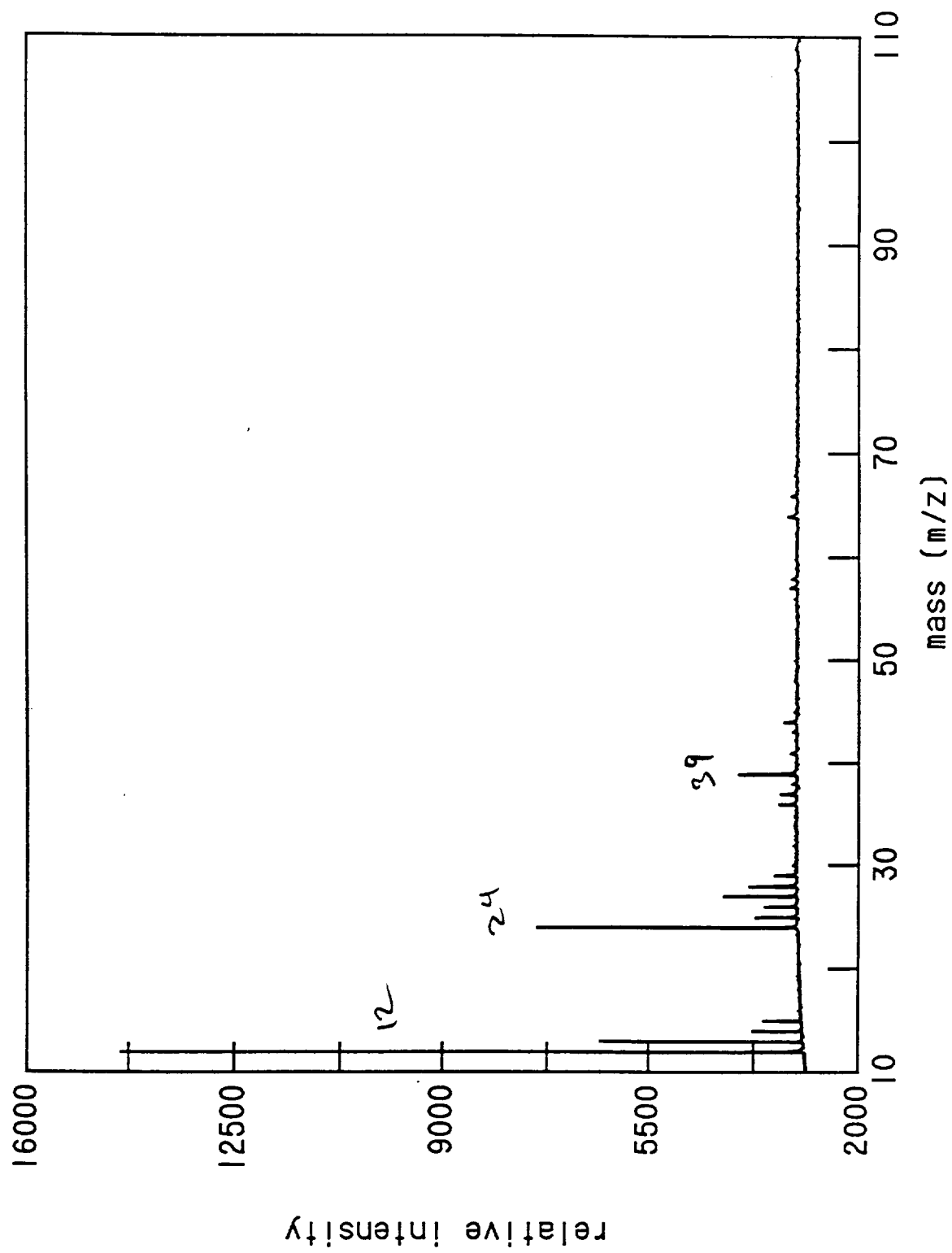
ORIGINAL PAGE IS  
OF POOR QUALITY



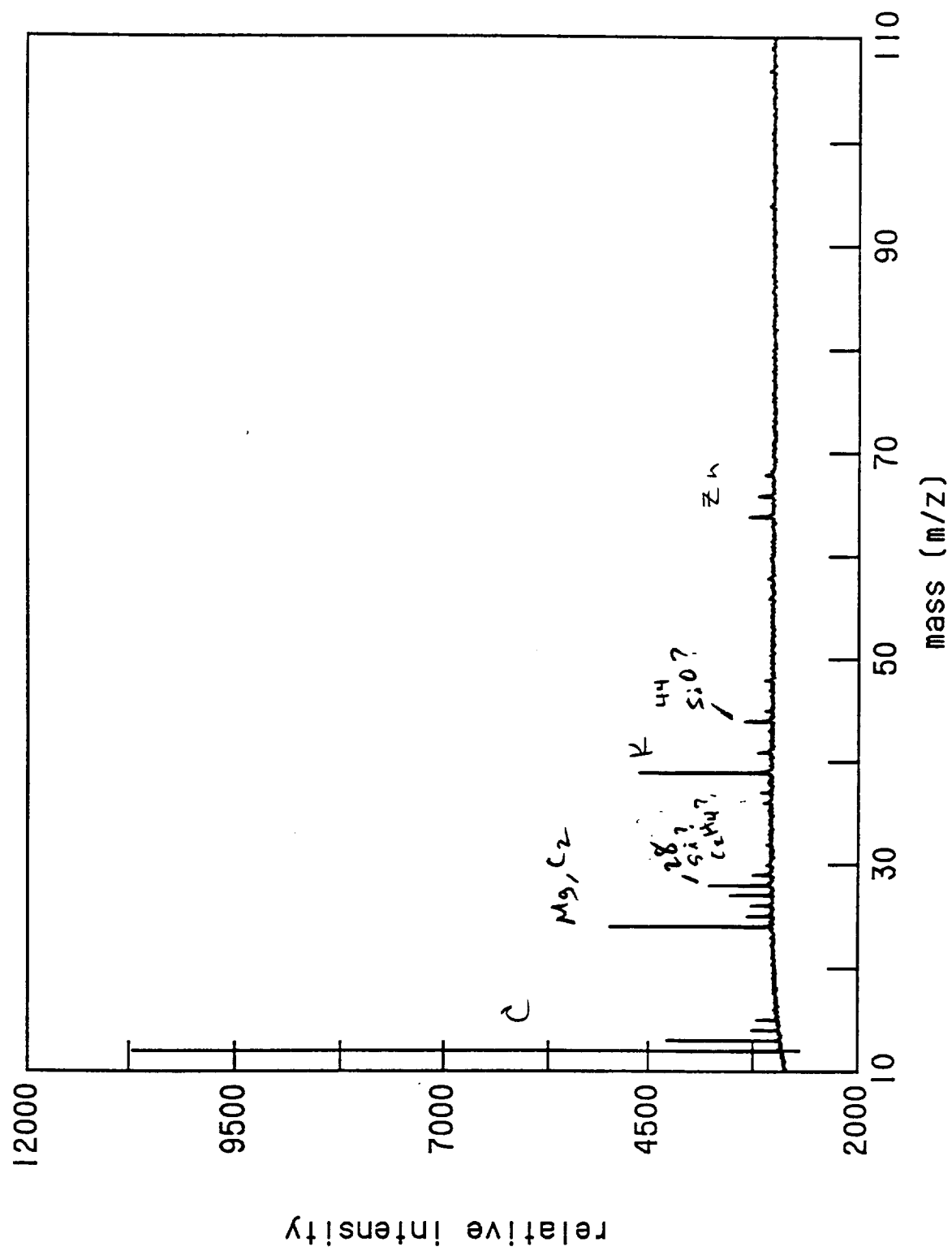
YB-1 LDEF 02-35 fe2508

266 nm

~2 1/2 ML erosion

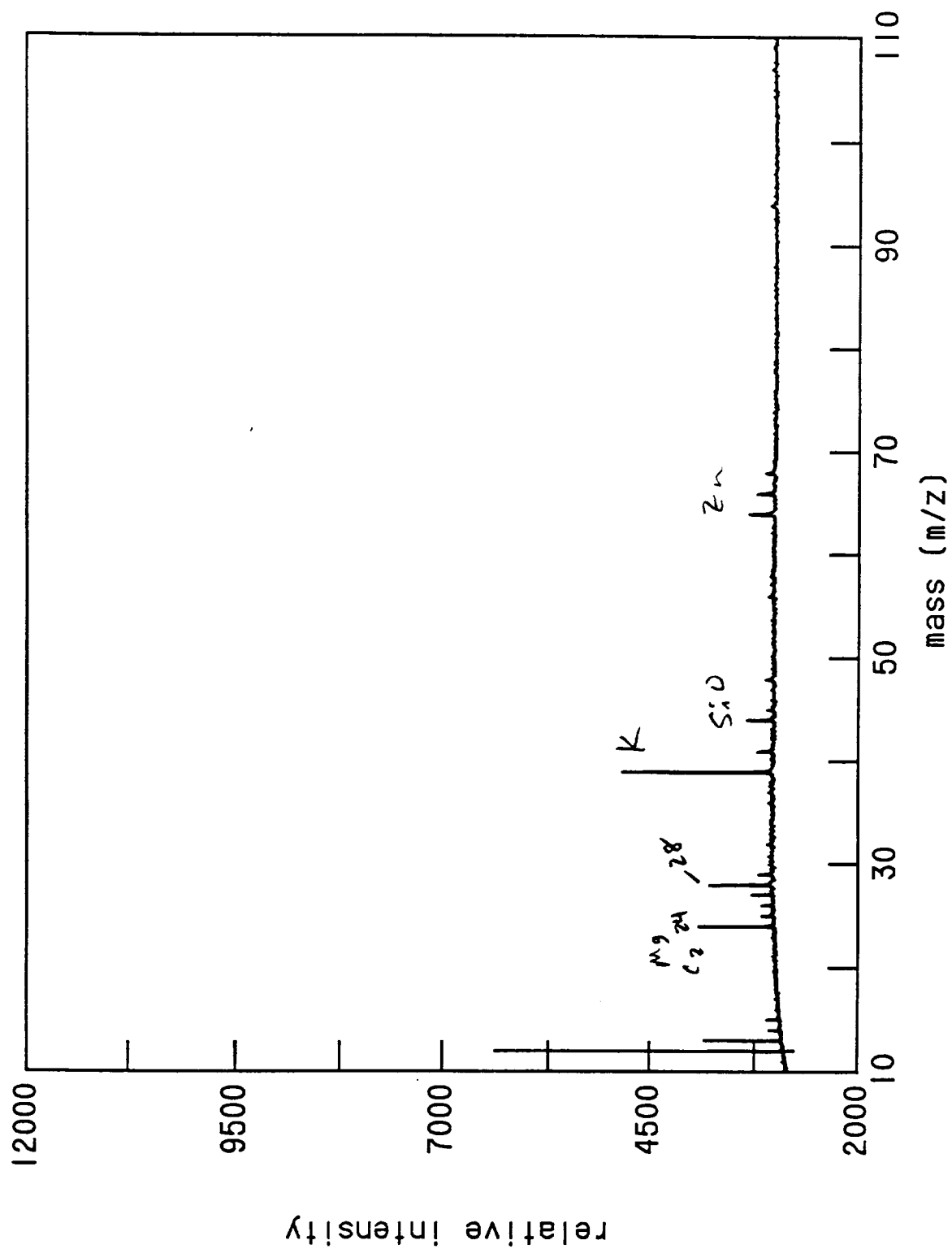


YB-1 control surface fe2510      266 nm  
static conditions



YB-1 control fe2511 266nm

$\sim \frac{1}{2}$  ML erosion

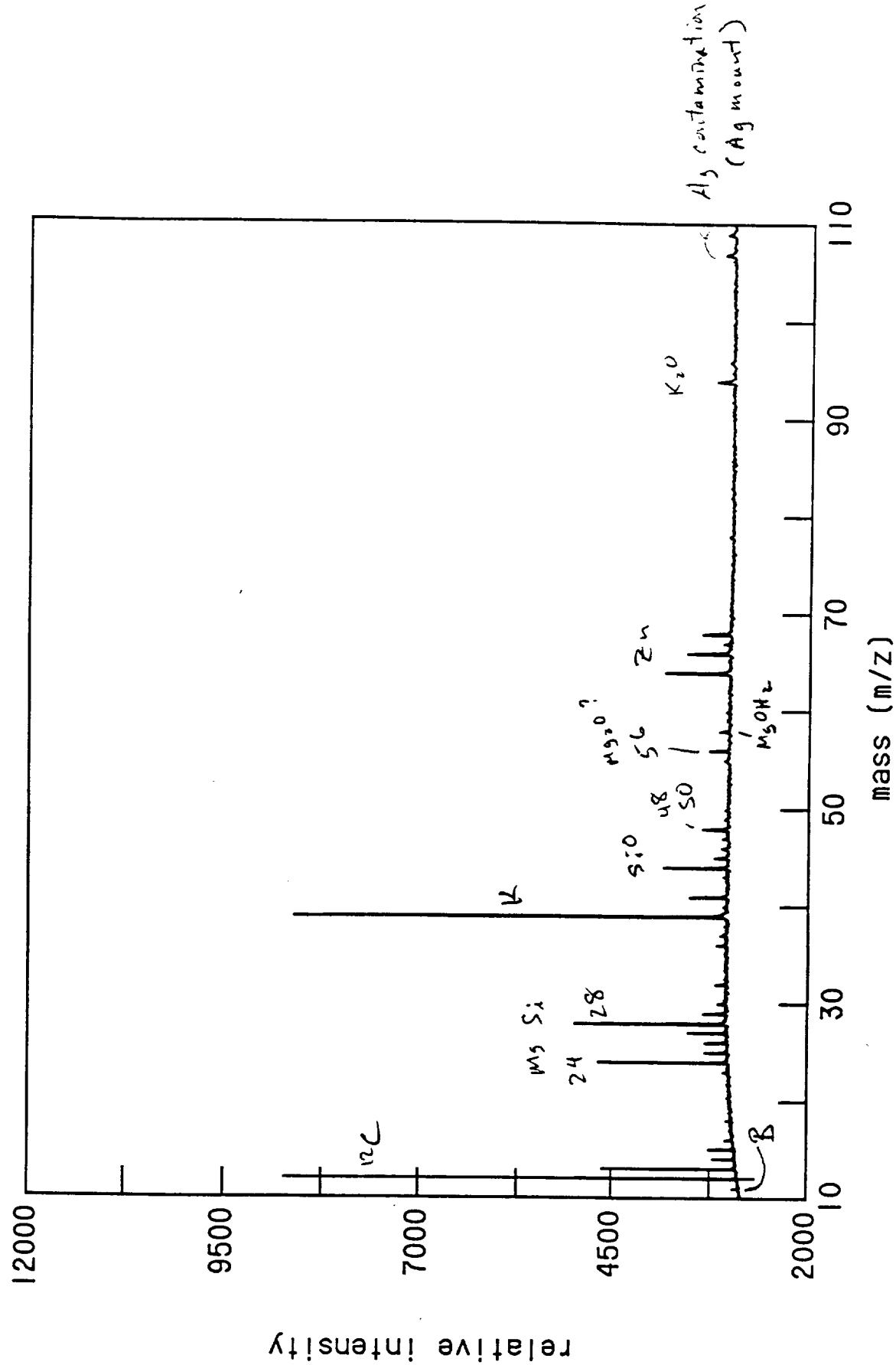


YB-1 control fe2512

266nm

~11% erosion

ORIGINAL PAGE IS  
OF POOR QUALITY



YB-1 control fe2513

266 nm

~2 1/2 ML erosion